

**DYNAMIC MODELING AND CONTROL OF REACTIVE DISTILLATION FOR
HYDROGENATION OF BENZENE**

A Thesis

by

OBANIFEMI ALUKO

Submitted to the Office of Graduate Studies of
Texas A&M University
in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

August 2008

Major Subject: Chemical Engineering

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Approved by:

Chair of Committee,	Juergen Hahn
Committee Members,	Mahmoud El-Halwagi
	Sergiy Butenko
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ABSTRACT

Dynamic Modeling and Control of Reactive Distillation for

Hydrogenation of Benzene. (August 2008)

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Chair of Advisory Committee: Dr. Juergen Hahn

This work presents a modeling and control study of a reactive distillation column used for hydrogenation of benzene. A steady state and a dynamic model have been developed to investigate control structures for the column. The most important aspects of this control problem are that the purity of the product streams regarding benzene need to be met. At the same time as little toluene as possible should be converted. The former is a constraint imposed by EPA regulations while the latter is tied to process economics due to the high octane number of toluene. It is required to satisfy both of these objectives even under the influence of disturbances, as the feed composition changes on a regular basis. The dynamic model is used for developing transfer function models of two potential control structures. Pairing of inputs and outputs is performed based upon the Relative Gain Array (RGA) and PI controllers were designed for each control structure. The controller performance was then compared in simulation studies. From our results, control structure 2 performed better than control structure 1. The main advantage of CS2 over CS1 is noticed in the simulation of feed composition disturbance rejection, where CS2 returns all variables back to steady state within 3 hrs while it take CS1 more than 20 hrs to return the temperature variables back to steady state.

DEDICATION

To my family and friends

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I am very grateful to my research advisor, Prof. Juergen Hahn for all the knowledge he provided and his direction in this project. His work ethic is one to be emulated. He made time available for his students and his timely response to inquiries were truly appreciated. I would also like to thank the other members of my committee, Prof. Sergiy Butenko and Prof. Mahmoud El-Halwagi, for their time and assistance during the course of this work. I would like to thank Ben Cormier for the initial help he provided in introducing me to Aspen Plus. I would like to thank the staff and professors in the Department of Chemical Engineering at Texas A&M University who helped during the whole period of my research.

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CHAPTER I

INTRODUCTION

Introduction to reactive distillation

Reactive distillation is process, where chemical reactions and separation are carried out in a single multifunctional process unit. As opposed to the conventional method used in the chemical process industries, in which the chemical reaction and the purification of the desired products are usually carried out separately and sequentially. This classic approach can be improved by the integration of reaction and distillation in a single column. This integration concept is called reactive distillation¹.

Such a configuration has several advantages which include higher selectivity, the heat of reaction being used to facilitate distillation by vaporizing the liquid phase, overcoming chemical equilibrium limitation, azeotropic mixtures being more easily separated than in a conventional distillation column. Also this integration reduces initial investment and operational cost by combining multiple units in one.

One of the most important industrial applications of reactive distillation columns is in the field of esterification such as the Eastman Chemical Company's process for the synthesis of methyl acetate². This process replaces a conventional flow sheet with 11 units to a single hybrid unit with a reactive and none reactive zone. With this process intensification, investment and energy cost were reduced by a factor of five³.

This thesis follows the style and format of *Journal of Physical Chemistry*.

Another important application of RD columns is in the preparation of ethers MTBE, ETBE and TAME which are produced in large amounts as fuel components⁴.

Despite the success of reactive distillation, it is important to know that the process is not always advantageous. For one part it may not be feasible for certain reactions and separation processes. Also, due to interaction of reaction and distillation in one single unit, the dynamic and steady state operational behavior can be very complex. As a result, the controllability and operability of this attractive process can be reduced.

Over the last two decades¹, especially after the commissioning of large scale plants for MTBE and methyl acetate production, reactive distillation has been used as a unit to fulfill several multiple chemical process objectives. Engineers and chemists have started looking beyond the classic esterification and etherification processes. Figure 1 shows methyl acetate separative reactor process by Eastman Chemical. Now RD columns have been successfully applied on a commercial scale to processes for hydrogenation, hydrosulfurization, isomerization and oligomerization.

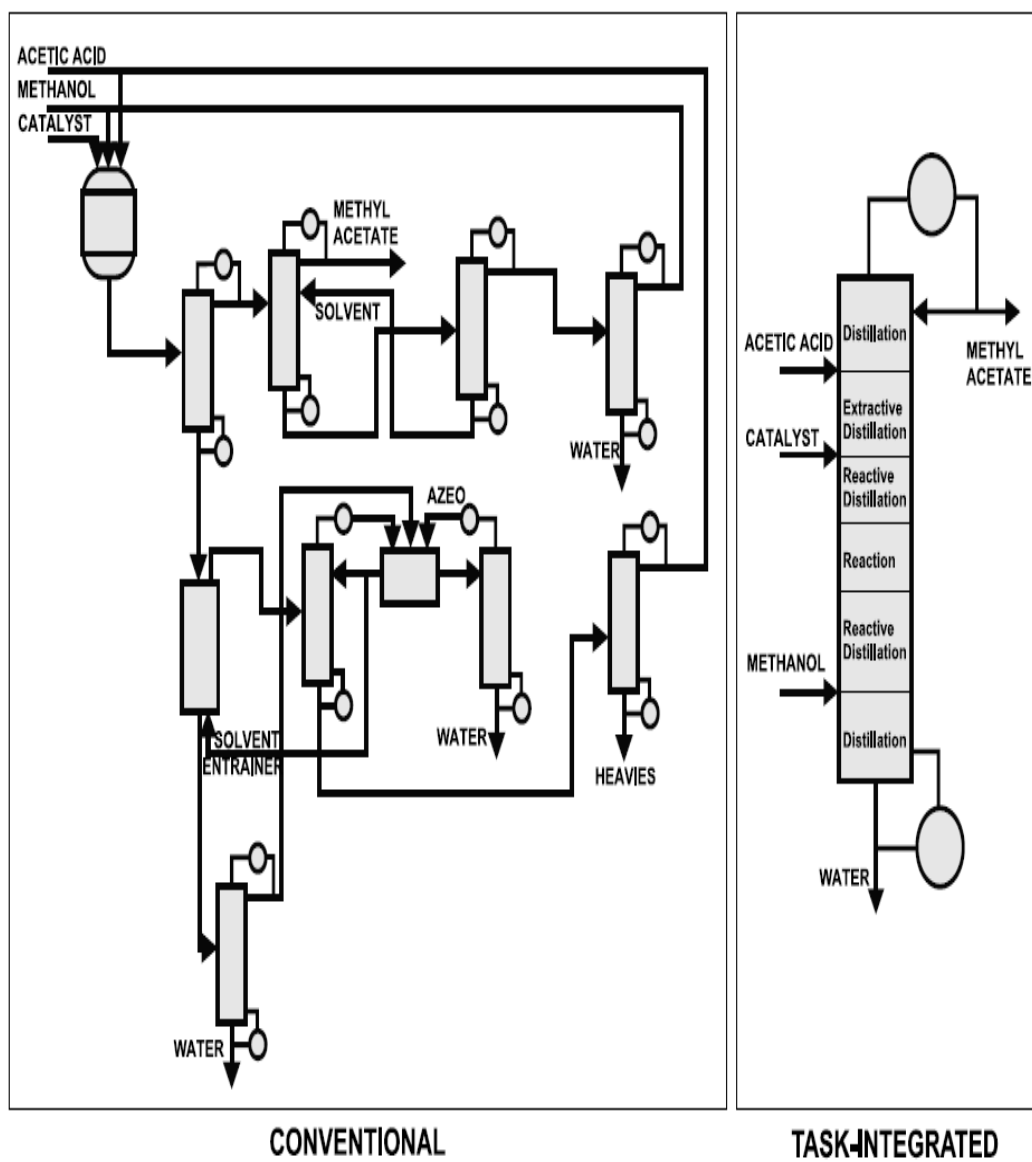


Figure 1: Plant integration in methyl acetate separative reactor process by Eastman Chemical (Adapted from⁵).

Another important area of its application is for the removal of small amounts of impurities to obtain high quality products like phenol. Reactive distillation can also be used for the recovery of valuable products like lactic acid, glycols, and acetic acids from waste streams.

Reactive distillation research greatly relies on the use of mathematical models. Initially, researchers developed models to describe the steady-state behavior of reactive distillation columns. Such a model, according to its underlying modeling assumptions, is classified as an equilibrium or a rate-based model. This classification usually refers to the treatment of liquid-vapor material and energy transfer mechanism. When thermodynamic equilibrium is assumed between the liquid and vapor phases, the model is an equilibrium one. Otherwise, a rate-based mechanism is employed to describe the material and energy transfer between the liquid and vapor phases. The chemical reactions also introduce a structural difference between the models. Indeed, the chemical reactions can be either described by some rate expressions or assumed to be at equilibrium. Sundmacher et al.⁶ presented an approach to characterize and classify a reactive distillation process according to its intrinsic physical-chemical behavior. Taylor and Krishna⁷ published a comprehensive review on reactive distillation that includes an extensive presentation of various models.

Benzene hydrogenation

Gasoline reforming is the process of altering the composition of gasoline to achieve a higher octane rating. Gasoline is a complex mixture of hydrocarbons, generally falling in the range of C₆-C₁₀, and different mixtures have different octane ratings. A reforming

process generally produces high octane aromatics one of which is benzene. Benzene is an undesirable carcinogenic impurity in gasoline, which is being regulated by the EPA due to studies showing a link between increased incidences of leukemia in humans exposed to benzene. Due to this, refining processes have to focus not only on producing high octane gasoline but also meeting the environmental standards, benzene reduction being one of the major regulations. Since most of the benzene is produced in the reformat stream, the benzene has to be removed downstream from the reformer. There are several processes used for the downstream removal of benzene, they are⁸:

- Alkylation.
- Reformate splitting and benzene extraction.
- Hydrogenation of benzene to cyclohexane.

The latter is the focus of this work. Hydrogenation in reactive distillation is a typical class of reacting system in which one of the components is non-condensable under the operating conditions¹. Hydrogenative distillation for the conversion of isophorone to trimethyl cyclohexane has been practiced since the 1960s. Recently many hydrogenation reactions have been investigated and commercialized successfully using reactive distillation.

The reactive distillation process for benzene hydrogenation developed by CR&L uses a supported nickel catalyst at high temperature and high pressure and offers several advantages apart from being highly selective. The process allows efficient contact of hydrogen and benzene, good temperature control and substantial removal of heat of reaction.^{9, 10} The down side to this method is the possibility of hydrogen taking part in undesirable reaction, for instance hydrogenation of toluene to methyl cyclo-hexane; this

is an unwanted reaction because toluene has a high octane rating and should remain a main component of the final gasoline mixture.

Process description and requirements

The Process being investigated is a reactive distillation column used for the hydrogenation of benzene. The column consists of 70 trays including a condenser and reboiler. The hydrocarbon feed enters the column above stage 45. The hydrogen required for the reaction has to be fed above the hydrocarbon feed stage and below the reactive zone. The hydrogen feed stage in this process is stage 40. There are three product streams coming out of the column; the distillate, the bottom product and the vent. The distillate is a liquid stream that contains the cyclohexane produced by the reaction between hydrogen and benzene. The bottom product is also a liquid stream containing the heavier keys in the hydrocarbon mixture and an insignificant amount of benzene. The vent is a vapor stream that contains most of the unreacted hydrogen. Since it is extremely difficult to condense hydrogen, any unreacted hydrogen has to be vented out and in some cases recycled and added to the original feed. A recycle stream is not included in our process because this would involve having another separation unit to separate the hydrogen from the lighter than light key hydrocarbons in the vent stream before recycling the hydrogen. This would involve investigating the dynamics of more than one column which is beyond the reach of this project.

The hydrogen feed has a flow rate of 2,000 lb/hr with pressure and temperature of 127 psi and 80F. The feed flow rate is 200,000 lb/hr at a pressure of 120 psi and a temperature of 270F. The reformate composition by weight is given in the table 1.

Table 1: Feed composition

Feed Components	Composition (weight)
Butane	0.01
Pentane	0.08
2,3-Dimethylbutane	0.01
3-Methylpentane	0.05
Hexane	0.03
Benzene	0.08
3-Methylhexane	0.02
2,4-Dimethylpentane	0.02
Heptane	0.01
Toluene	0.30
Xylene	0.22
Cumene	0.17

The reactive zone is between stage 6 and 20 and is filled with catalyst required to hydrogenate the benzene. The reactive zone was chosen to take advantage of the difference in volatilities of the feed components. It is above reformat feed stage because benzene is one of the light keys of the feed compositions therefore it flows upward towards the reactive zone to react with hydrogen which also flows up the column. The heavy keys flow downward and therefore do not go through any reactions.

There are two reactions that take place in the reactive zone:

- The hydrogenation of benzene to cyclohexane, the wanted reaction.
- The hydrogenation of toluene to methylcyclohexane, which is an unwanted reaction.

The Latter is an unwanted reaction because toluene contributes to a high octane number for the stream. Even though it is possible for reactions to occur outside the reaction zone, it happens at a very slow rate without a catalyst that its effects can be ignored. It is therefore important that the reactive zone is not placed immediately above the reformat

feed stage because a substantial amount of the toluene would enter the reactive zone and can get saturated.

The rates of reaction of the two reactions are assumed to be first order with respect to each reactant. They are expressed as follow:

- $r_1 = k_1 C_{BZ} P_{H_2}$

- $r_2 = k_2 C_{Tol} P_{H_2}$

They are both exothermic reactions.

Figure 2 shows the conventional method of a distillation column followed by a reactor.

The column diagram is given in Figure 3.

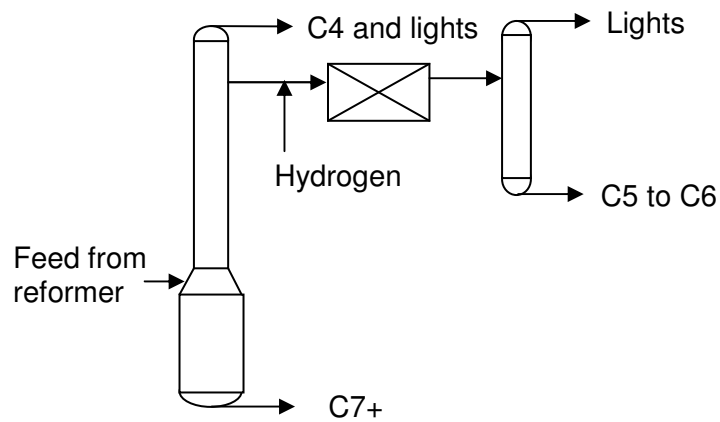


Figure 2: Regular distillation column with reactor (conventional method)

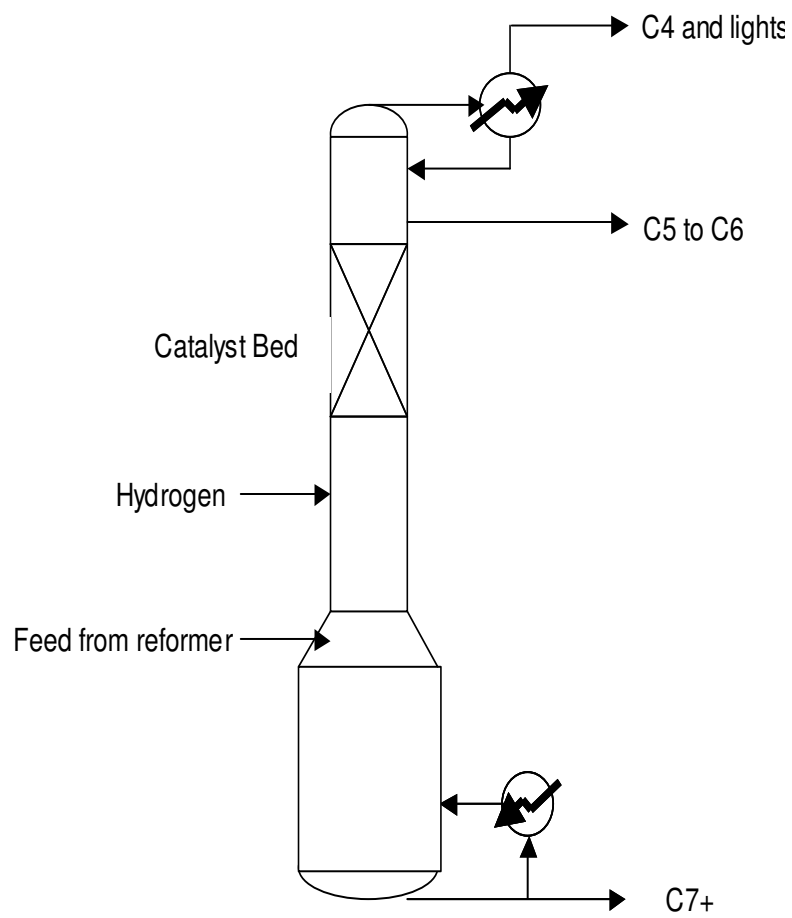


Figure 3: Process diagram

Objective

The main objective of the column under investigation is to hydrogenate benzene without hydrogenating the toluene. The exit concentration of the benzene in the light reformat stream should be less than 1% weight to meet EPA regulations and the toluene recovery in the bottom should be 99.5%. A control scheme is to be devised to accommodate disturbances to the process. The main disturbance associated with this process is the feed composition of the hydrocarbon feed. The benzene varies from 3% to 15% weight fraction while the heavier components can range from 40% to 80%. Due to these disturbances, it takes a while for the column to reach a new steady state and the product may deviate largely from its product specification. Appropriate controllers can return the process to a new steady state faster after disturbances and can also control the product specification. To design our controller we require a dynamic model, so the objectives of this work is to develop a dynamic model for our process, design controller to be implemented in the process and test the controllers in response to disturbances in the system.

Previous work

Reactive distillation processes can result in an economically attractive alternative to conventional process designs, where reaction and separation are carried out in different processing units¹. Consequently, there has been a lot of interest in this type of process intensification in recent years. Emphasis has been on steady-state modeling and the foundations of RD column design. Still, comparably little work has been done on dynamics and control of a reactive distillation process.

Unlike regular distillation processes, reactive distillation systems usually have two objectives:

- Final product purity; and
- Desired level of reactant conversion.

Sneesby et al.¹¹ consider these two requirements and develop a two-point control scheme based upon linear PI controllers for a 10 stage ETBE reactive distillation column. The authors identified a tray temperature as a controlled variable to regulate the product composition and chose an inferential model to predict the reactant conversion.

Additionally, general recommendations for the control of this type of process are given.

^{12, 13, 14, 15} Tade and co-workers extensively investigate catalytic distillation columns for the production of ETBE. Their work includes research on input multiplicity¹², conversion inferences from temperature measurements¹⁶, and predictive control¹⁷. Bock et al.¹⁸ present a control scheme consisting of two independent feedback loops for the production of isopropyl myristate. However, even for small disturbances in the acid feed rate, the controllers were unable to achieve acceptable performance, presenting a need for feed forward control. Kumar and Doutidis¹⁹ studied the control structure of a reactive distillation column with three controlled variables. The process was the formation of ethylene glycol from the reaction of ethylene oxide and water. This work was extended to ethyl acetate reactive distillation afterwards by Vora and Daoutidis²⁰. Lextrait²¹ worked on 5 X 5 control structure of a TAME packed reactive distillation column with PI controllers.

Al-Arjaf and Luyben conducted extensive case studies on control structure selection and controller design for different reactive systems²² via simulations. These included the

control of an ideal two-product reactive distillation column, of a methyl acetate reactive distillation column, of an ethylene glycol trayed reactive distillation column, control for ETBE synthesis and pentene metathesis. Their control structures generally consisted of conventional linear feedback controllers coupled with ratio control for ensuring that a sufficient amount of both reactants would be fed to the column.

So far there is no literature on the control of a benzene hydrogenation RD column. Despite the fact that this is a process that is being used in chemical plants all over the world, controlling the process has received little research. This shows that there exist a need for further study of the dynamic and control of a RD column for the hydrogenation of benzene.

CHAPTER II

DEVELOPMENT OF DYNAMIC MODEL FOR REACTIVE DISTILLATION

Modeling is a cornerstone of science and engineering. Human knowledge relies greatly on the use of mathematical modeling to conceptualize reality. Usually, a scientist or engineer postulates some physical mechanisms, writes a mathematical model and then validates it. Any solvable mathematical model that represents the underlying assumptions is usually sufficient for developing an understanding of the physical mechanisms. However, from a control perspective, some representations of models are more attractive than others.

This chapter discusses concepts related to developing both steady state and dynamic models, with assumptions and simulation relevant to the work presented here.

Steady state model

A steady state model of a distillation column can either be rate-based or equilibrium-based. In an equilibrium-based model it is assumed that the bulk vapor and the bulk liquid phase are in chemical equilibrium to each other¹⁰. This means that the vapor and liquid stream exiting from such control volumes will be in equilibrium to each other. There will be no temperature gradient within the region where the equilibrium assumption is valid. In a rate-based model, however, the liquid and vapor interface are assumed to be in equilibrium. There is a temperature gradient in the phases and mass transfer takes place between the bulk and the interface of two phases.

The justification of using an equilibrium-based model in building a distillation column model has often been questioned in many articles²³. The fact that the streams leaving a tray are never in equilibrium to each other has initiated the use of efficiency in equilibrium-based model. However the difficulty and the uncertainty associated with determining the efficiency of each tray is also a concern. Lee et al.²⁴ compared simulation result of an equilibrium-based and non equilibrium based model of a multicomponent reactive distillation column. The conclusion drawn was to prefer generalized non equilibrium model for the simulation as opposed to an equilibrium based model because of the difficulty associated with the prediction of tray efficiencies. Later Taylor et al.²⁴ compared the two approaches and pointed out that “with ever increasing computing power these simulations are not only feasible, but in some circumstances they should be regarded as mandatory”. In another study however Rouzineau, Prevost and Meyer²⁵ showed that by taking reasonable values for the Murphee efficiencies one can get similar simulation results in equilibrium models and rate-based models. They also pointed out that if obtaining reasonable Murphee efficiencies is difficult then it will also be problematic to predict some of the rate-based model parameters. While the critical factor is to obtain a good description of vapor-liquid equilibrium most real distillation columns, both trayed and packed, can be modeled via stage equilibrium models.²⁶

Our steady state model was derived using an equilibrium based column modeled in Aspen Plus²⁷. The physical properties of the streams were computed using the Peng-Robinson equation of state. The steady state model is the first step to creating a dynamic model.

Assumptions for the model

As discussed in the last chapter making the right assumptions are important for generating a model with desired properties. The assumptions for modeling the steady state column were chosen to be:

- Equilibrium based model.
- Ideal gas law to describe gas properties.
- Murphee efficiency has been assumed to be equal to 1.
- The reaction rate constant follows an Arrhenius equation.

Another major assumption made in the simulation was to exclude the reaction that involves the hydrogenation of toluene. This assumption was made under the notion that most of the toluene would be found at the bottom of the column, with only a negligible amount entering the reactive zone, hence the toluene would not be able to react with the hydrogen.

The process was simulated to meet certain product specifications:

- The benzene weight concentration is less than 1% in the light reformat stream (distillate product).
- The toluene recovery is 99.5% in the bottom product.

The simulation results from the steady state run are given in the table 2:

Table 2: Steady state results of benzene hydrogenation

	Distillate	Bottom	Vent
Temperature (F)	65.04	449.53	65.04
Pressure (Psia)	115	135.6	115
Phase	Liquid	Liquid	Vapor
Mass Flow rate (lb/hr)	60000	140000	2000
Butane	0.02924	0.00000	0.12271
Pentane	0.25692	0.00000	0.29249
2,3-Dimethylbutane	0.03277	0.00000	0.01686
3-Methylpentane	0.16441	0.00000	0.06778
Hexane	0.09893	0.00000	0.03202
Benzene	0.00000	0.00014	0.00000
Cyclohexane	0.28476	0.00000	0.06628
3-Methylhexane	0.06221	0.00179	0.00844
2,4-Dimethylpentane	0.06615	0.00001	0.01472
Heptane	0.00450	0.01235	0.00046
Toluene	0.00000	0.42857	0.00000
Xylene	0.00000	0.31429	0.00000
Cumene	0.00000	0.24286	0.00000
Hydrogen	0.00010	0.00000	0.37823

The results closely match steady state data of the plant provided by CD Tech. The results show that the percentage by weight of benzene in the distillate is less than 1%, and the mass fraction of toluene in the bottom is 0.42857, which is 99.9% toluene recovery, basically no toluene is found in the distillate. Once the steady state requirements are met, the next step is to create a dynamic model.

Dynamic model

Mathematical models for dynamical systems are particular sets of equations that represent certain behaviors. Among the different approaches the modeling of chemical processes, Hangos and Cameron²⁸ have presented a formal strategy to systematically handle systems modeled according to first principles. The models are defined and processed according to the modeling assumptions, which truly define a behavior. The

formal representation of the modeling assumptions enable the authors to deal with complex chemical process models having a certain structure. The procedure stresses that an engineering model has to be understood as a set of algebraic equivalent models, when models are defined through equations. This idea of great significance, since one realizes that a mathematical model, a set of differential and/or algebraic equations, is one of many possible representations of a behavior.

As stated earlier, from the steady state model, an assumption on the type of model is made on whether the distillation column is going to be rate-based or an equilibrium-based model. Other assumptions will have to be made which would have an effect on how numerically solvable the model will be. Since the dynamic model is a set of differential algebraic equations (DAE), inappropriate assumption may lead to a model not solvable due to index problems.

The index problem was first identified by Petzold (1982), followed by Gear (1988). The problems of solving of a dynamic process model arises with DAE of index 2 or higher. Brennan, Campbell and Petzold²⁹ pointed out, that the numerical solution of these types of systems has been the subject of intense research in the past few years. While dealing with high index DAE systems has been a topic of intensive research since index problem was first determined, it is possible to completely avoid this problem by proper modeling and nobody wants a high index DAE system in the first place.

Moe in his Ph.D. dissertation³⁰ presented his study on modeling and index reduction. His work mainly focused on formulating solvable process models and manipulating models into a more manageable form. A modeling method for developing low index models was presented along with two index reduction algorithms. In the same year Moe,

Hauan, Lien and Hertzberg³¹ applied a modeling method for modeling a system having both phase and reaction equilibrium which guaranteed the resulting model to be semi-explicit index one. They also looked into the initialization methods of the DAE system. Using a systematic modeling approach, Ponton and Gawthrop³² have shown how to formulate sets of differential and algebraic equations, which are of index one for describing certain classes of chemical processes. Following simple rules, the modeler can represent the dynamical systems as DAE's of index one. The work presented here also ensured to use the set of assumptions so that the resulting system is not higher index. The approach taken by them suggests that the extensive balance equations should be combined to eliminate interphase flow variables. Hence, by "intelligent" modeling, equilibrium assumptions also lead to DAE systems of index one.

Below is a summary of the key ideas to be taken under consideration when modeling a system:

- A mathematical model seen as a set of DAE's is only a particular representation of a behavior.
- Different representations of a particular model originate from a unique set of modeling assumptions
- Different representations of a particular model exhibits can exhibit different numerical, control or modeling-related properties.

Aspen Dynamics³³ was used to study the dynamics and control of the process. The steady state results from Aspen Plus were exported to Aspen Dynamics to create the dynamic model. Additional specifications had to be added to the steady state model to this dynamic model. The RD column has a diameter of 11.5ft to accommodate the vapor

rates in the column and a weir height of 0.31ft. Simple tray stages were used in the simulation with an 8000lb liquid holdup in the reactive zone, with residence time of approximately 2 min.

Disturbance response without controller

Simulations were carried out for disturbances in the system to investigate how the system responded to these changes without controllers being present. The disturbances charged to the system were: changes in the feed flow rate, changes in the feed temperature and changes in the feed benzene composition. The responses are illustrated below.

Change in feed flow rate

Figure 4 shows the response of several variables in the column in response to disturbance in the feed flow rate without the control structures being implemented.

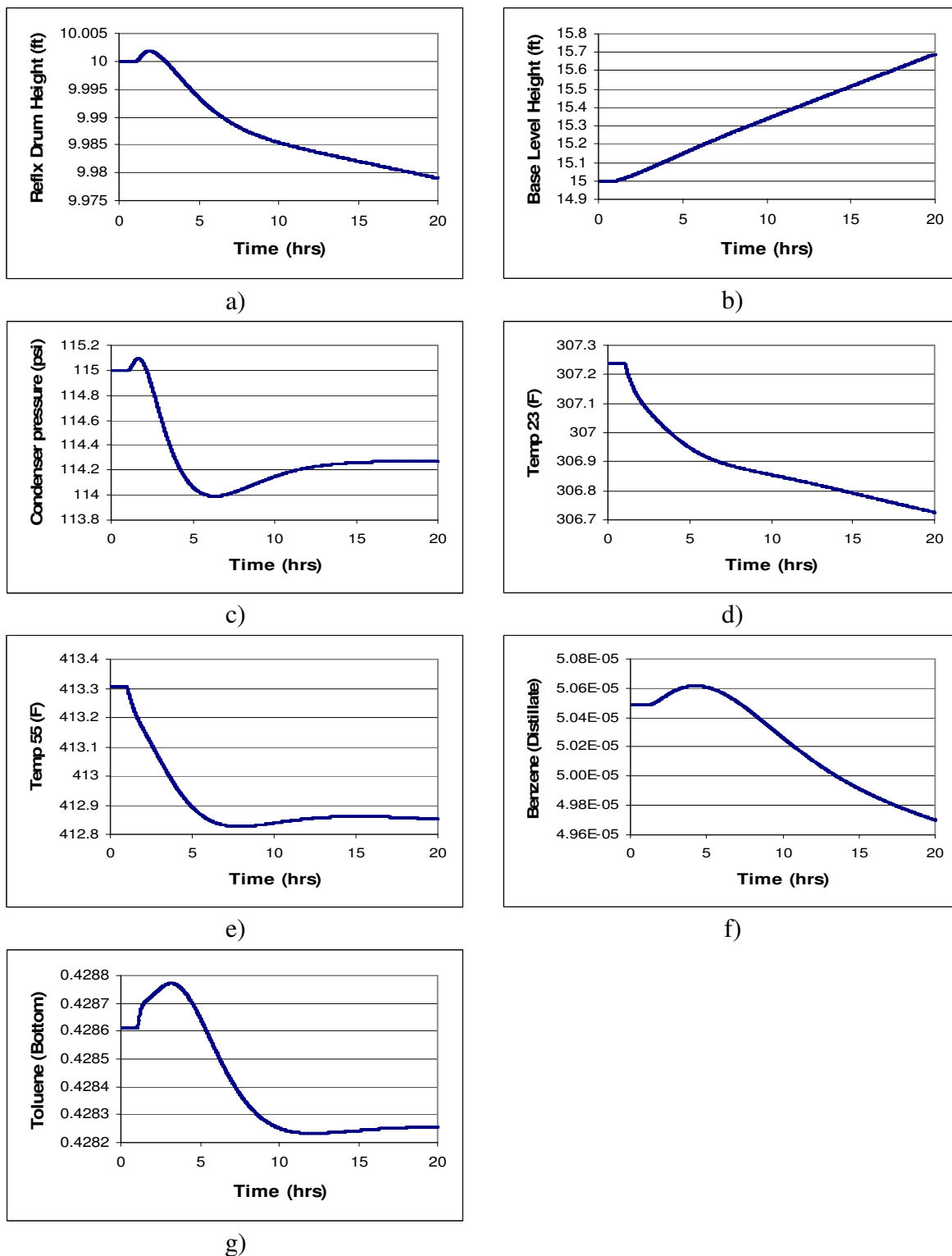


Figure 4: Disturbance in feed flow rate. Response of: a) reflux drum liquid height b) base level liquid height c) condenser pressure d) temperature on stage 23 e) temperature on stage 55 f) Benzene composition in distillate g) Toluene composition in bottom

Figure 4 shows that a disturbance in the feed flow rate leads to the process moving away from its steady state, and some variables moving towards a new steady state. The base level liquid keeps increasing until it starts flooding, at which point, the model loses its validity.

Change in feed temperature

Figure 5 shows the response of several variables in the column in response to disturbance in the feed temperature without the control structures being implemented.

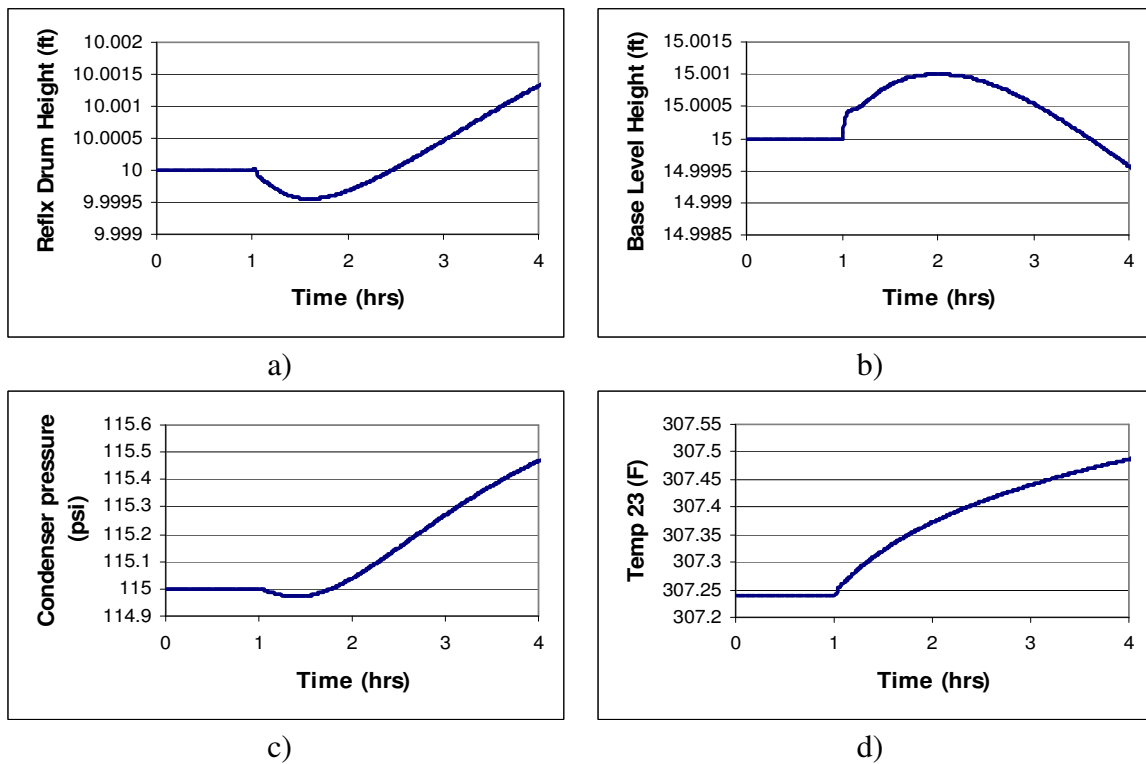


Figure 5: Disturbance in feed temperature. Response of: a) reflux drum liquid height b) base level liquid height c) condenser pressure d) temperature on stage 23 e) temperature on stage 55 f) Benzene composition in distillate g) Toluene composition in bottom

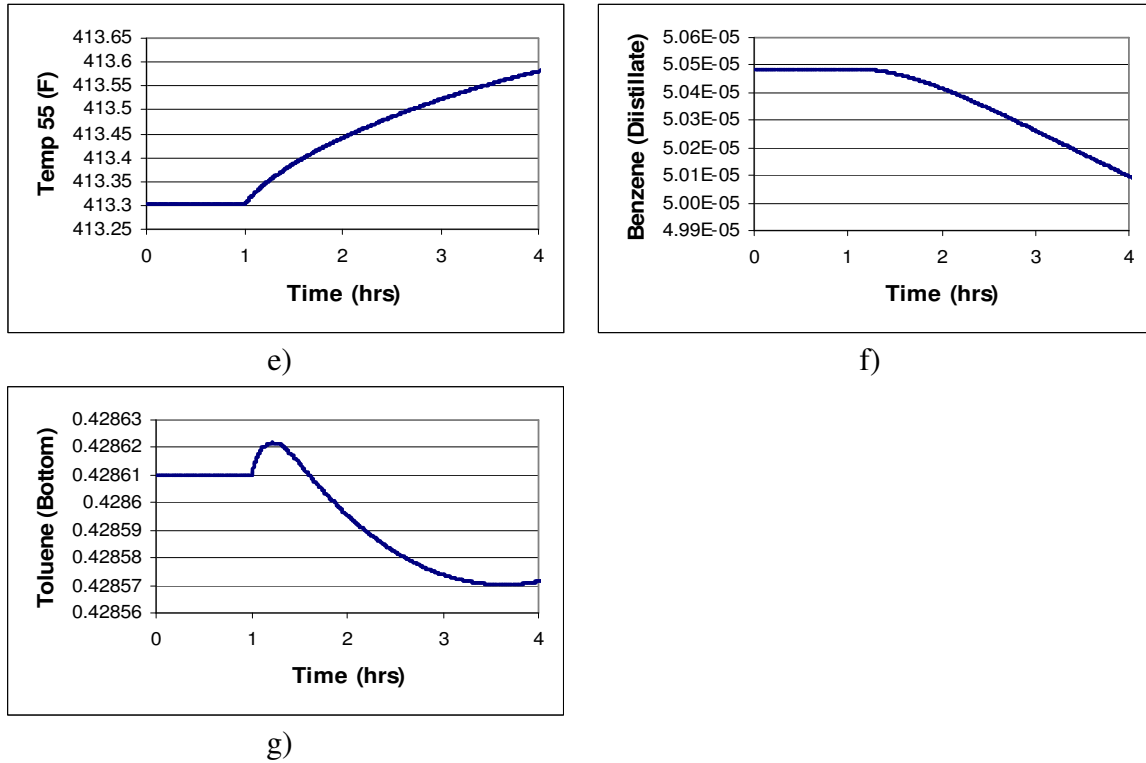


Figure 5: Continued

Disturbance in the feed temperature also demonstrates that the system moves away from steady state. The simulation results ended a little after 4 hours, this was because the simulator had convergence problems after the disturbance was charged at the 1 hour mark. But the results presented are just used to illustrate that the system moves away from steady state after feed temperature disturbance.

Change in feed composition

Figure 6 shows the response of several variables in the column in response to disturbance in the feed benzene composition without the control structures being implemented.

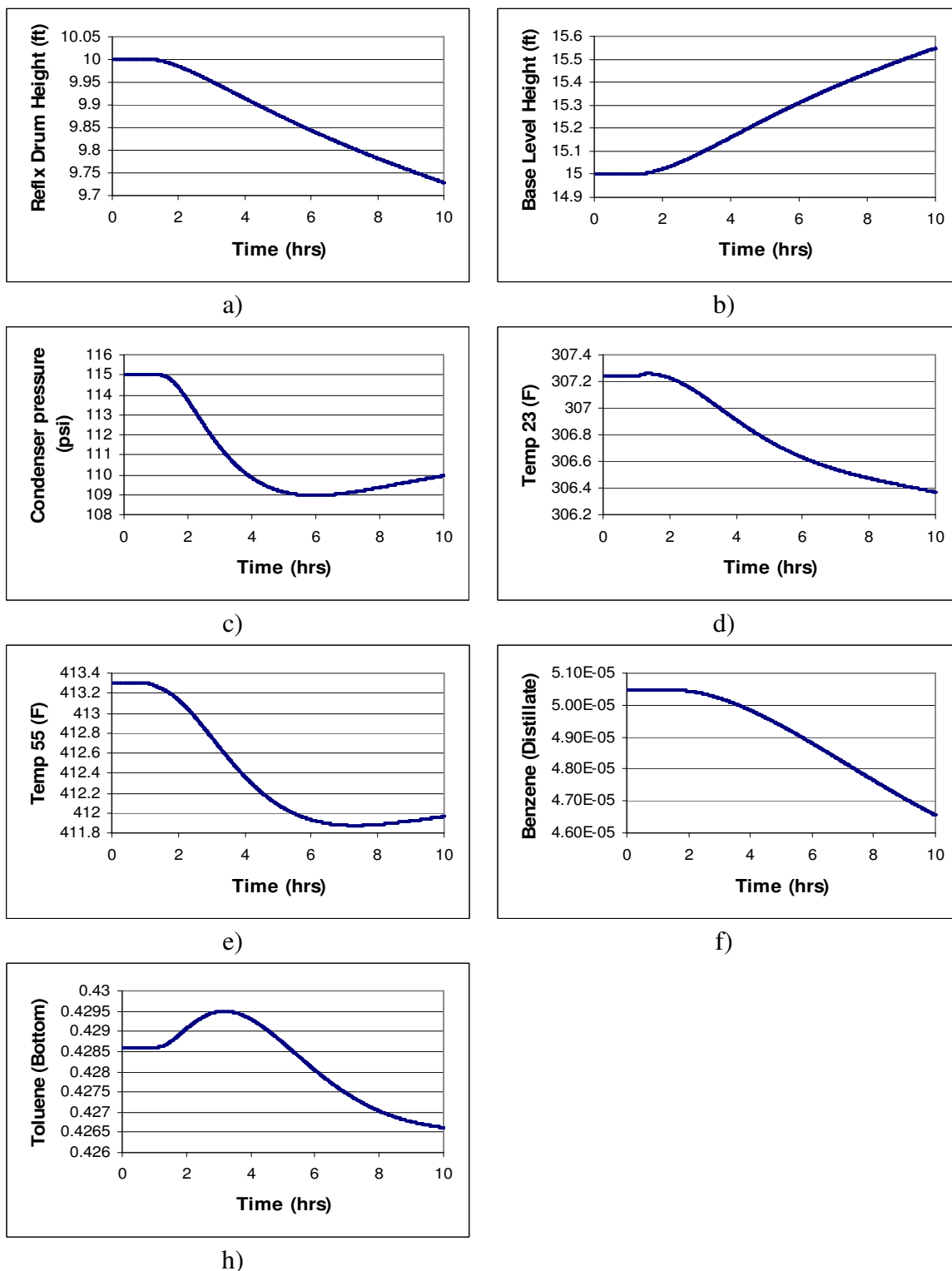


Figure 6: Disturbance in feed benzene composition. Response of: a) reflux drum liquid height b) base level liquid height c) condenser pressure d) temperature on stage 23 e) temperature on stage 55 f) Benzene composition in distillate g) Toluene (bottom)

Figure 6 also indicates that the system deviates from its steady state after a disturbance in the feed benzene composition.

Discussion

From the simulation results, we observe that these three types of disturbances cause the process to deviate from its steady state values and it takes several hours before it reaches its new steady state. This further emphasizes that a control system is necessary to return the process to steady state within a considerable amount of time.

CHAPTER III

CONTROL OF A REACTIVE DISTILLATION COLUMN

Overview

Distillation is probably the most studied unit operation in terms of control. Control of distillation columns refers to the ability of keeping certain variables at or near their setpoints whenever there is a disturbance or set point change in the plant. Many papers and books have been devoted to the investigation and exploration of different aspects of distillation column control over the last half century³⁴. Usually, the control of reactive distillation columns presents several difficulties due to the existence of multiple steady states³⁵. Output multiplicities have direct implications for the operability and controllability of the reactive distillation¹⁴. Minor perturbation in feed conditions or disturbances may produce a transition to a less favorable steady state. Sneesby et al.¹⁵ investigated different strategies to control the transition from an undesirable steady state to a desirable one in a MTBE reactive distillation column. Sneesby et al.¹⁶ published recommendations for the control of an ETBE reactive distillation column.

Barlett³⁶ studied, via dynamic simulation, different control strategies for a MTBE reactive distillation column, and highlighted possible limitations of traditional feedback control. A hybrid feedforward-feedback controller using molar feed ratio control was proposed. However, the author suggested that advanced control strategy (predictive control) would have its benefits.

Gruner et al³⁷ described a nonlinear control scheme for continuous reactive distillation. They designed a robust observer based on temperature measurements that was

incorporated into a nonlinear state feedback control scheme. Jimenez and Costa-Lopez³⁸ analyzed the simulation, modeling, and control of a butyl acetate reactive distillation. The authors described the process as an equilibrium stage model. They presented an efficient decentralized control scheme with PID controllers.

Al-Arfaj and Luyben^{39, 40, 41, 42} conducted extensive case studies for different reactive systems. First they investigated the control of an ideal two-product reactive distillation column⁴¹. The process was a two-feed 30-tray reactive distillation column. Four components constituted the chemical system. A single reversible reaction occurred inside the column involving two reactants and two products. The authors employed dynamic equilibrium model to investigate six control alternative structures. In subsequent publication⁴⁰, the authors investigated the control of a methyl acetate reactive distillation column.

Balasubramhanya and Doyle III⁴³ considered the production of ethyl acetate via batch reactive distillation. They proposed a nonlinear control algorithm based on a reduced nonlinear model, which captured the essential dynamic of the process.

The control of batch or semi-batch reactive distillation has been studied in a number of communications.^{44, 45, 46, 47, 48, 49} Sorensen et al.^{50, 51} extensively studied the optimal control of batch reactive distillation.

The procedure for determining which process variables should be controlled by manipulating certain values is called is control strategy design⁵². Dynamic simulations can be used to provide a picture of how the plant will behave when there is a set point change and disturbances. Controller system design can be broken into following steps:

- Formulate control objective

- Identify controlled and manipulated variables
- Choose a control strategy and structure
- Specify controller settings.

The control objective can generally be formulated based on safety concerns, environmental regulations, and economic objectives. In this particular work it is mainly driven by environmental regulations and economic considerations. The first step involved in designing a control strategy for a distillation column is choosing the controlled and manipulated variables. The variables are then paired based on their level of interaction between the variables; this is achieved by performing relative gain array analysis. PI controllers are then tuned to be implemented in the control structure. The settings of the controllers are obtained using IMC tuning rules, the closed loop structure is tested to investigate the control performance and identify if further tuning would be required. The controllers are tuned until a satisfactory control performance is observed.

Choosing controlled and manipulated variables

Choosing a set of controlled and manipulated variables is the first step taken for developing a plant wide control structure for a process. To do this, the degree of freedom of the process must first be determined; this is the number of variables that can be controlled. Knowing the degree of freedom of a process is useful so that you do not try to over or under control the process.

The mathematical approach to finding the degree of freedom is to subtract the number of independent equations from the total number of variables. An easier approach is to simply add the total number of rationally placed control valves.

For instance, in a simple distillation column with a single feed, distillate and bottom products, we have 5 control valves, one for each of the following streams: distillate, bottom, reflux, condensing and heating medium. So this column would have 5 degrees of freedom.

Inventories on any process must be controlled²³, these include liquid levels and column pressure. For the case of the simple distillation column, the liquid level in both the reflux drum and base of the column and the column of the pressure must be controlled. Subtracting these three variables that must be controlled from 5, leaves us with two degrees of freedom. Therefore, we are left with 2 additional variables that can be controlled in this simple distillation case.

The remaining two variables chosen to be controlled depend on the specifications of the process and the control resources provided for the process. Some common situations include: controlling composition of the light-key impurity in the bottoms and the composition of the heavy-key impurity in the distillate or controlling a temperature in the rectifying section of the column and a temperature in the stripping section of the column. Once all controlled variables are determined, we still have the problem of deciding what manipulated variable to use to control what controlled variable. This “pairing” problem is solved by performing a relative gain array (RGA) analysis which would be discussed later.

Open loop step test

Once the controlled variables and manipulated variables are chosen, open loop tests are performed on the column. Open loop tests are simulation runs on the dynamic column

carried out without any controllers in place. It can be called the natural response of the column to changes in the manipulated variables. The responses of the controlled variables to changes in the manipulated variables are recorded. This information is used to determine the range in which the linear fit is appropriate and it is also used to provide the relationship between the input and output through transfer functions.

Linearity of the model

In order to determine the transfer function relationships between input and output, step changes in the input/manipulated variables in both directions and of different magnitudes are simulated and the response of the output variables are used to determine the range in which changes in the manipulated variables can be fit by linear relationships. For instance, for similar changes in the manipulated variable in opposite directions, should produce responses that are approximately mirror images of each other. In this case fitting a linear transfer function would be appropriate. However if the two responses are completely different in nature from each other then this is not a good assumption. For large changes in the manipulated variables such a case can often be observed. Another way to judge this could be to determine if the superposition principle holds in the response of the controlled variables. For example for a step change of magnitude 4, the response has to be the same as that of the addition of the two different responses with change of magnitude 1 and change of magnitude 3 of the manipulated variable for a linear model. Also, if a step change of magnitude 2 produces a response of magnitude 2, a step change of twice the initial magnitude (i.e. magnitude of 4) should

produce a response of twice the initial response and so on. This step identifies the range in which fitting the transfer functions will be valid.

Transfer function

The transfer function is an expression which dynamically relates the input and the output in a process model. $Y(s) = G(s) U(s)$ where Y is the output, U is the input and G is the transfer function relating them. So if a transfer function is known between one input and one output, the change in the output can be computed for a change in the input. One important property of the transfer function is that one can calculate the steady-state change in output given a change in input by directly setting $s = 0$ in $G(s)$. Another important property of transfer functions is that they can be added. A single process output variable can be influenced by more than one input variable. The total output change is calculated by summing up the changes of the output if only one of the inputs were changed at a time.

$$Y_i(s) = \sum_j G_{ij}(s) U_j(s)$$

Where, $Y_i(s)$ is the i^{th} controlled variable, $U_j(s)$ is the j^{th} manipulated variable and G_{ij} is the transfer function between the i^{th} controlled variable and the j^{th} manipulated variable.

Relative Gain Array (RGA) analysis

Relative gain array was introduced by Bristol⁵³. Bristol developed a systematic approach for the analysis of multivariable process control problems. The approach is convenient because it requires only the process gain matrix K and provides two important pieces of information⁵⁴:

1. A measure of process interactions.
2. A selection criteria for the most effective pairing of controlled and manipulated variables.

Consider a process multivariable process with n manipulated variables and n controlled variables. The relative gain between a controlled variable y and a manipulated variable u is defined as the dimensionless ratio of two gains, the open-loop gain and the closed loop gain:

$$\lambda_{ij} = \frac{(\partial y_i / \partial u_j)_u}{(\partial y_i / \partial u_j)_y} = \frac{\text{gain}(\text{open-loop})}{\text{gain}(\text{closed-loop})}$$

From the equation above, the numerator is a partial derivative with all the manipulated variables held constant except u_j . This is the open-loop gain (K_{ij}) between y_i and u_j .

Similarly the denominator is evaluated with all of the control variables held constant except y_i . This is the closed-loop gain that indicates the effect of u_j on y_i when all other variables are held constant.

The relative gain is then arranged in a relative gain array (RGA) as shown below:

$$\Lambda = \begin{matrix} & u_1 & u_2 & u_3 & u_4 \\ \begin{matrix} y_1 \\ y_2 \\ \dots \\ y_3 \end{matrix} & \begin{bmatrix} \lambda_{11} & \lambda_{12} & \dots & \lambda_{1n} \\ \lambda_{21} & \lambda_{22} & \dots & \lambda_{2n} \\ \dots & \dots & \dots & \dots \\ \lambda_{n1} & \lambda_{n2} & \dots & \lambda_{nn} \end{bmatrix} \end{matrix}$$

There are five ranges of value which an element in the RGA can have:

1. $\lambda_{ij} = 1$. In this situation the closed-loop and open-loop gains between y_i and u_j are identical. This is an ideal situation; it follows that y_i and u_j should be paired.
2. $\lambda_{ij} = 0$. This indicates that the open-loop gain y_i and u_j is zero, therefore u_j has no effect on y_i and they need not be paired.
3. $0 < \lambda_{ij} < 1$. The closed-loop gain is larger than the open-loop gain. Within this range, the interaction between the two loops is greatest when $\lambda_{ij} = 0.5$.
4. $\lambda_{ij} > 1$. In this situation, closing the loop reduces the gain between y_i and u_j .
Thus, the control loops interact. As λ_{ij} increases, the degree of interaction increases. When λ_{ij} is very large, it is impossible to control multiple outputs independently.
5. $\lambda_{ij} < 0$. When λ_{ij} is negative, the open-loop and closed-loop gains between y_i and u_j have opposite signs. It follows that y_i and u_j should not be paired because the control loops interact by trying to “fight each other”^{55, 56} and the closed-loop system may become unstable.

The overall recommendation from RGA analysis is to pair the controlled and manipulated variables so that corresponding relative gains are positive and as close to one as possible.

Tuning of controllers

After the transfer functions are derived and optimal pairing between the controlled variable and manipulated variable are found, the controller can be tuned. First we choose the type of controller, either P, PI or PID controller. Then the setting of the controllers is tuned to get good control performance. PI and PID controller settings can be determined by a number of alternative techniques. The Internal Model Control (IMC)⁵⁷ method is one of the most commonly used methods in practice. Other methods of designing controllers are using the Direct Synthesis (DS)⁵⁸ method, On-line tuning and other tuning relations based on Integral Error Criteria, i.e. IAE (Integral Absolute Error), ISE (Integral squared error) or ITAE(Integral time-weighted absolute-error)⁵⁹.

When the controller has been tuned, it is then implemented and the control performance is investigated.

CHAPTER IV

CONTROLLER DESIGN

Manipulated and controlled variables

In this process, we have six degrees of freedom: bottom flow rate, distillate flow rate, vent flow rate, condenser duty, reboiler duty and reflux flow rate. We have the choice of manipulating these six variables to control six other variables, we use the conventional 5X5 control structure²³, leaving one manipulated variable free. In our case, it is possible to have two 5X5 control structures (CS1 and CS2), where the manipulated variable which was not used in CS1 is used to substitute one of the manipulated variable initially used, this creates the second control structure, CS2.

The manipulated variables for both control structures are given below:

For CS1,

- Bottom flow rate, distillate flow rate, vent flow rate, reboiler duty and condenser duty.

For CS2,

- Bottom flow rate, distillate flow rate, vent flow rate, reboiler duty and reflux flow rate.

The controlled variables chosen are the same for both control structures. The liquid levels in both the reflux drum and base level are chosen to be controlled; the other three variables are the pressure in the column, temperature in the stripping section and temperature in the rectifying section.

Since there are 70 stages in the column, we have to decide the two stages to place the two temperature controllers. The first stage chosen for temperature control is a stage below the reactive zone (stage 23) and the second stage to be controlled is a stage a quarter way up the column from the reboiler (stage 55).

Since we have chosen five manipulated variables and five controlled variables for both control structures, RGA analysis is then used to determine which manipulated variable would be used to control a chosen controlled variable.

Linearity of the model

In order to design the controller the transfer function fit should be done in a region where linear relationship holds between the manipulated and controlled variables. As discussed earlier, this means that the two responses obtained for the same controlled variable for a given change in the manipulated variable in opposite direction should be approximately mirror image of one another. The figures below show that the model exhibits close to linear behavior since mirror images are derived for equal changes in positive and negative directions. Two cases are presented by the figures below: The effect of changing the bottom flow rate by $\pm 0.2\%$ and the effect of changing the reboiler duty by $\pm 0.05\%$. Figures 7 and 8 show the response to changes in bottom flow rate and reboiler duty respectively.

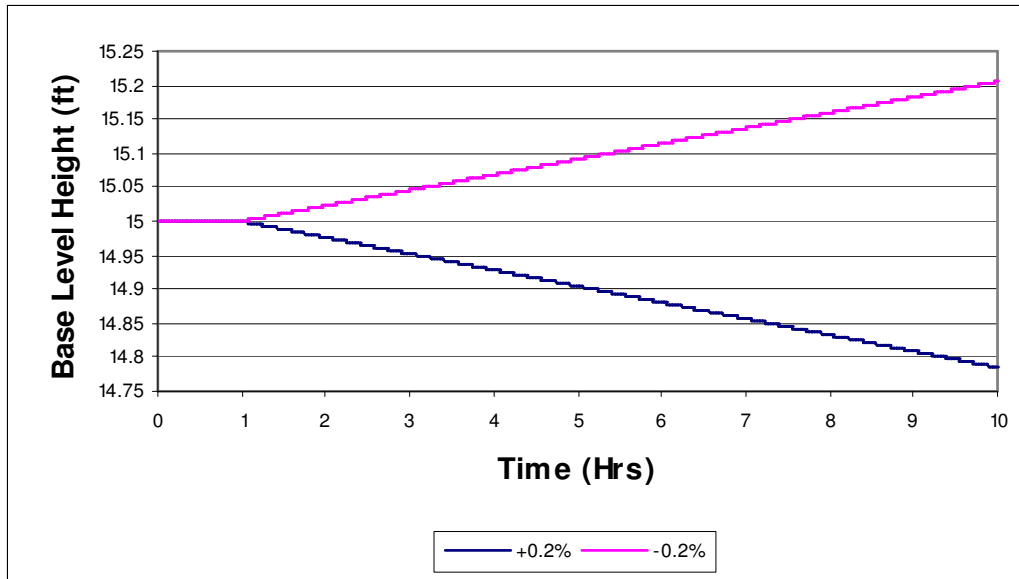


Figure 7: Response of base level height to changes in bottom flow rate

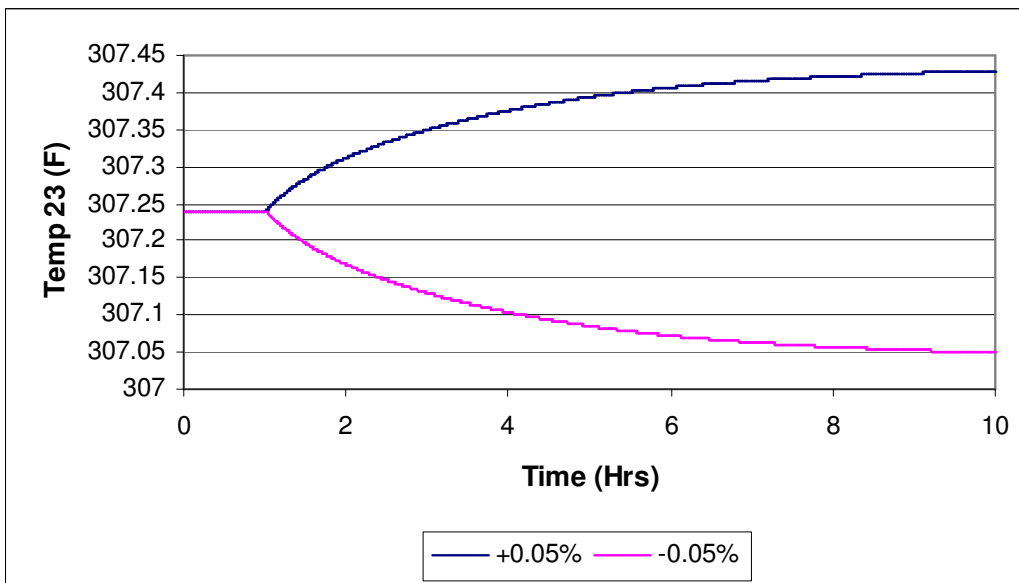


Figure 8: Response of temperature on stage 23 to changes in reboiler duty

The responses of other controlled variable to changes in the manipulated variables give similar results, showing the linearity of the model.

Transfer functions

We also perform open loop tests to obtain transfer functions between manipulated variables and controlled variables. A transfer function is an expression which dynamically relates the input and the output of a process model. $Y(s) = G(s) U(s)$ where Y is the output, U is the input and G is the transfer function relating them. Accordingly if a transfer function is known between one input and output, the change in the output can be computed for a change in the input. In this particular study there are five input variables which are the manipulated variables and five output variables which are the controlled variables. As a result of this a 5 X 5 control structure there will have 25 transfer functions since each combination of input-output variables results in one transfer function. From our simulations, we obtained responses to step changes in the manipulated variables and from the figures we obtained the transfer functions.

Figure 9 shows the response of the five controlled variables to change in bottom flow rate.

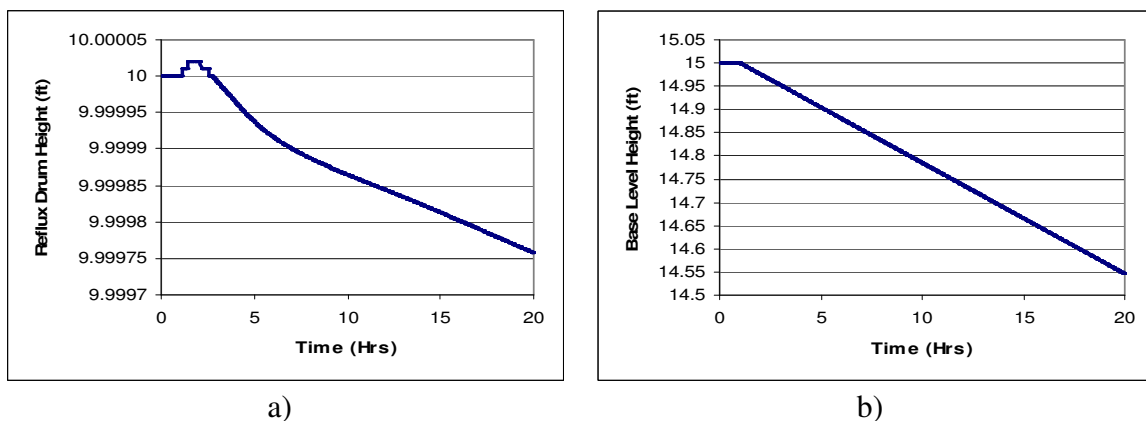


Figure 9: Change in bottom flow rate. Open loop response of: a) reflux drum liquid height b) base level liquid height c) condenser pressure d) temperature on stage 23 e) temperature on stage 55

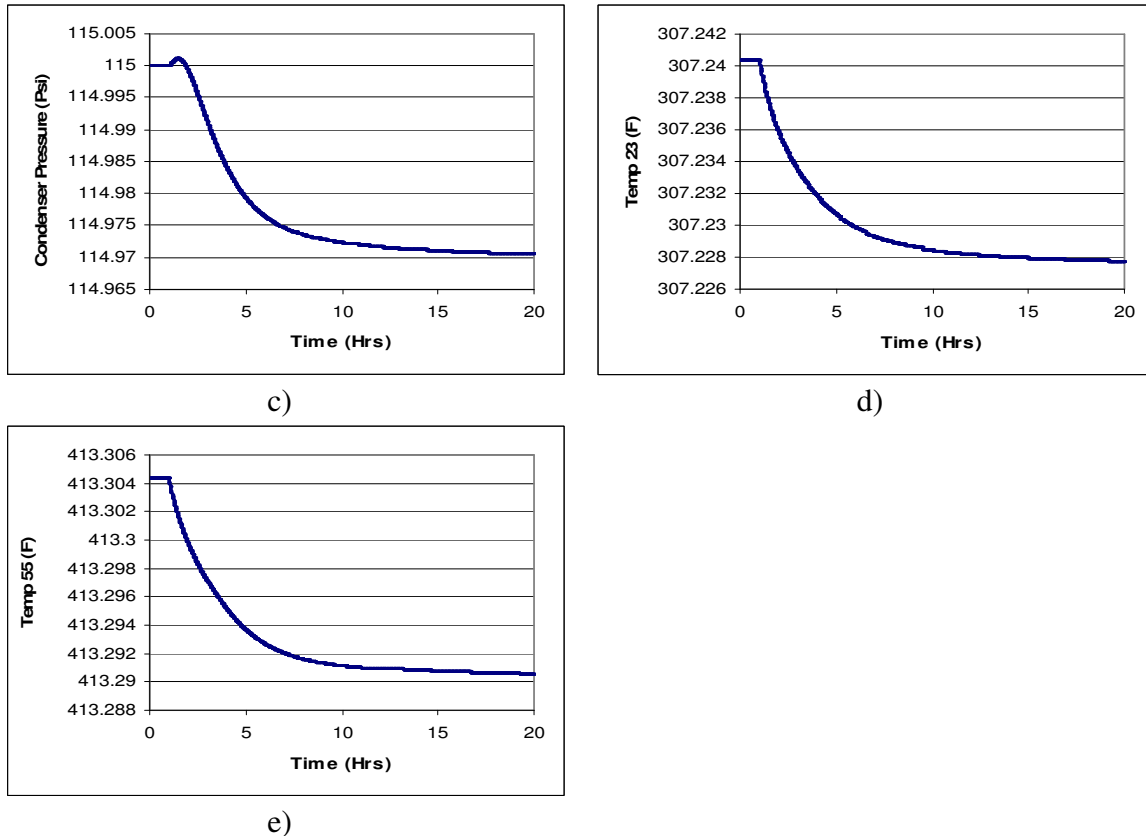


Figure 9: Continued

Figure 9 shows the transfer function profile for the five measured variables to changes in the bottom flow rate. From the figure above, we can see that a change in the bottom flow rate has an effect on each controlled variable. But each response varies in regards to the extent on which it is affected by the bottom flow rate. The reflux drum and base level liquid height respond as an integrating process, i.e. the process increases with time and does not reach a steady state. If allowed to continue over a long period of time, the two liquid drums will start drying up. The condenser pressure, temperature on stage 23 and 55 has a first-order process response. As it can be seen in the figure 9, the three latter variables reach a new steady state. Also we see that there are variables not really affected by the bottom flow rate change compared to other variables. The base level liquid height

change is much more than the change in the reflux drum's liquid height. Also the pressure and temperature do not change much in magnitude.

Figure 10 shows the response of five controlled variables to a change in distillate flow rate.

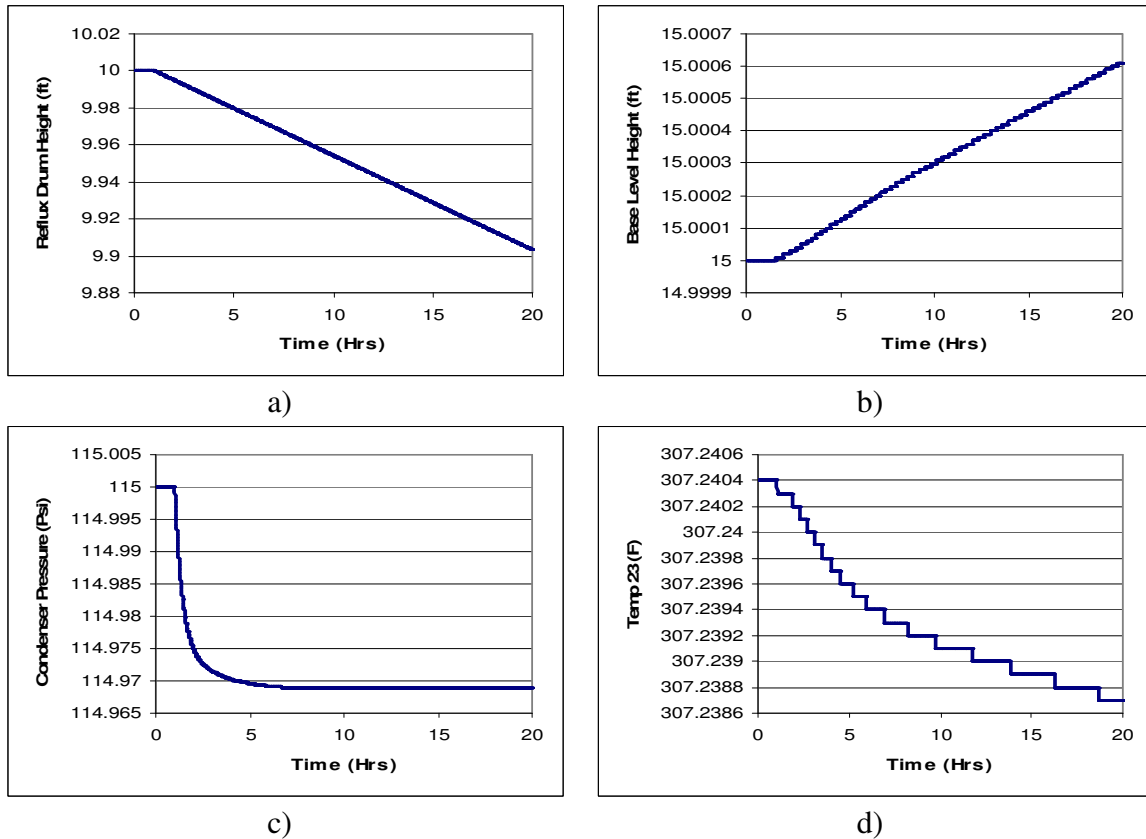
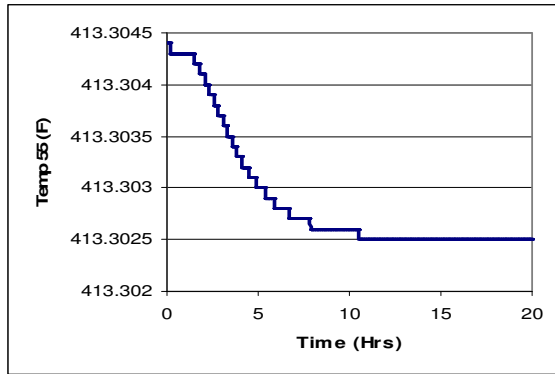


Figure 10: Change in distillate flow rate. Open loop response of: a) reflux drum liquid height b) base level liquid height c) condenser pressure d) temperature on stage 23 e) temperature on stage 55



e)

Figure 10: Continued

The distillate flow rate change also gives an integrating process response for both reflux drum and base level liquid height. An increase in the distillate flow rate causes the reflux drum liquid level to reduce, if the process is let to run for a long period of time, the reflux drum would dry up while the base (reboiler) drum would flood. The other three variables respond with a first-order process. Changes in the pressure, temperature and base level liquid height are negligible compare to the change in the reflux drum liquid height. Some of the changes are so small that the profile appears as steps, these steps are numerical problems and not physical. We can infer that the distillate flow rate would be used to control the reflux drum liquid height, but we leave assumptions for analysis that would actually give optimal pairing of the variables in the process. This would be achieved by RGA analysis.

Figure 11 shows the response of five controlled variables to change in vent flow rate.

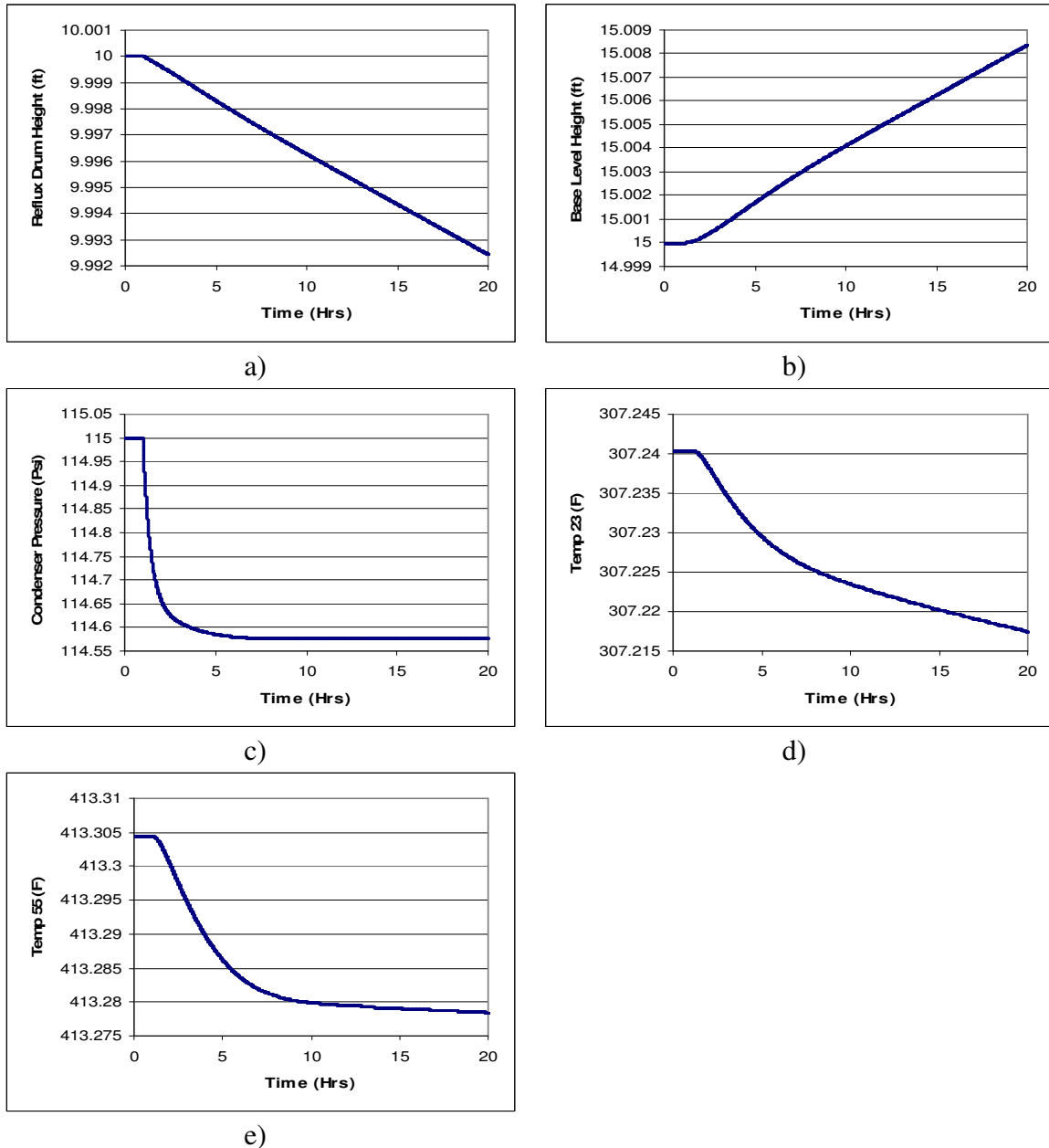


Figure 11: Change in vent flow rate. Open loop response of: a) reflux drum liquid height b) base level liquid height c) condenser pressure d) temperature on stage 23 e) temperature on stage 55

We would expect a change in the vent flow rate to have a visible effect on the pressure of the column, since it means either reducing or increasing the amount of vapor in the column. Figure 11 shows that increasing the vent flow rate, letting more vapor out of the

system, would reduce the condenser pressure. This reduction in pressure is so far the largest out of the three manipulated variables noticed, and has the fastest response. The temperatures on both stages studied are reduced but not profoundly, and the two liquid levels show an integrating process response.

Figure 12 shows the response of the five controlled variables to a change in the reboiler duty.

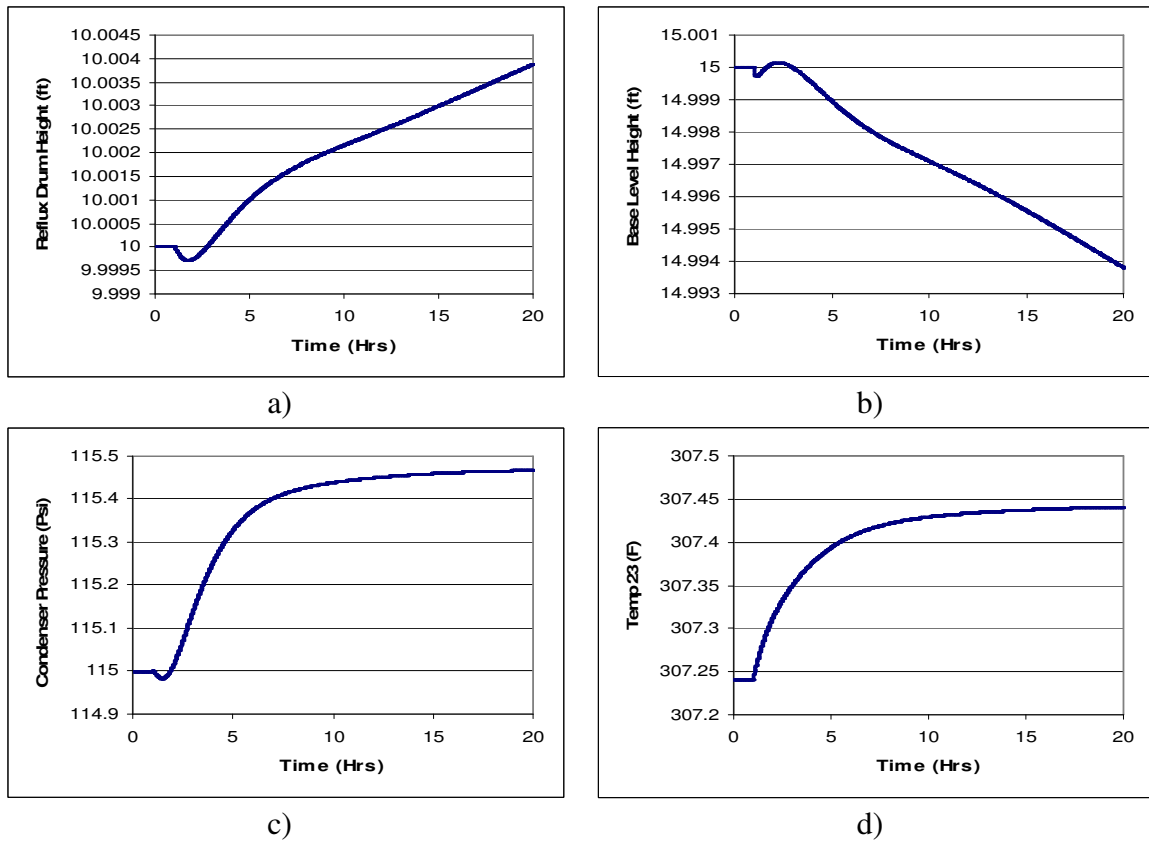
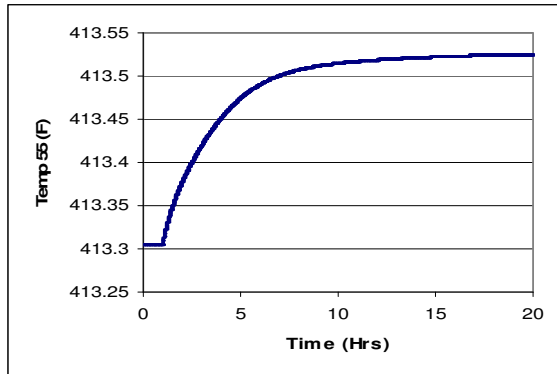


Figure 12: Change in reboiler duty. Open loop response of: a) reflux drum liquid height b) base level liquid height c) condenser pressure d) temperature on stage 23 e) temperature on stage 55



e)

Figure 12: Continued

An increase in the reboiler duty in the column, increases the temperature in the column, produces more vapor, and hence increases the column pressure. It boils more liquid in the base level drum, so the liquid height reduces and if let to continue for a long period of time, dries up. These are behaviors that are expected, but there are some aspects of the column we can not explicitly state without performing the simulation and observing how they respond, but from figure 12, we see that the liquid height in the reflux drum increases as the reboiler duty increases.

Figure 13 shows the response of the five controlled variables to a change in the condenser duty.

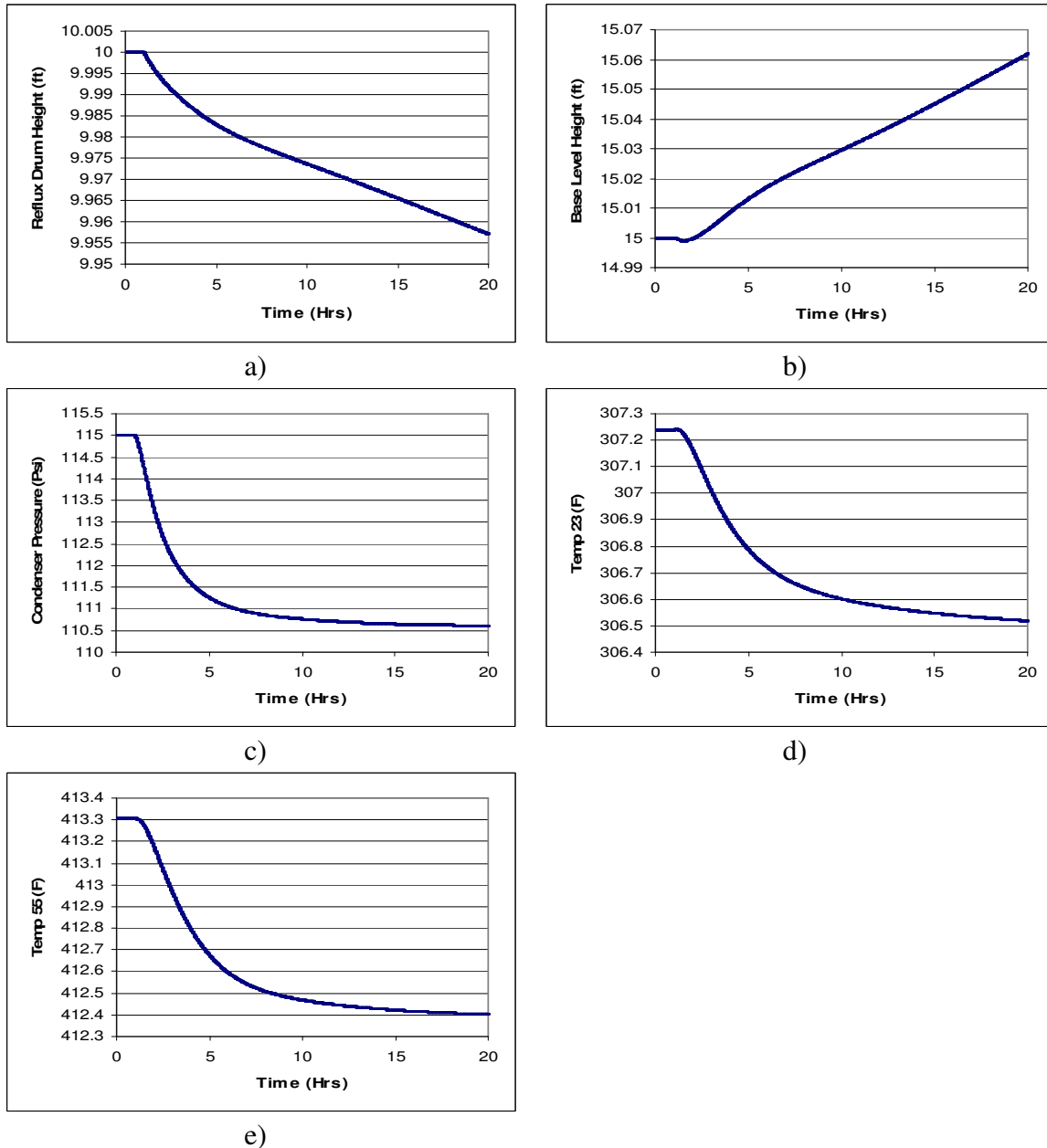


Figure 13: Change in condenser duty. Open loop response of: a) reflux drum liquid height b) base level liquid height c) condenser pressure d) temperature on stage 23 e) temperature on stage 55

Increasing the condenser duty, reduces the temperature in the column, also condenses more vapor, therefore reducing the vapor in the column, hence reducing the pressure. It has an integrating-process response on both liquid levels in the column. Changes in the

condenser duty and reboiler duty show that these manipulated variables have more of an effect on the temperature on both stages than the first three manipulated variables studied.

Figure 14 below shows the response of the five controlled variables to a change in the reflux flow rate.

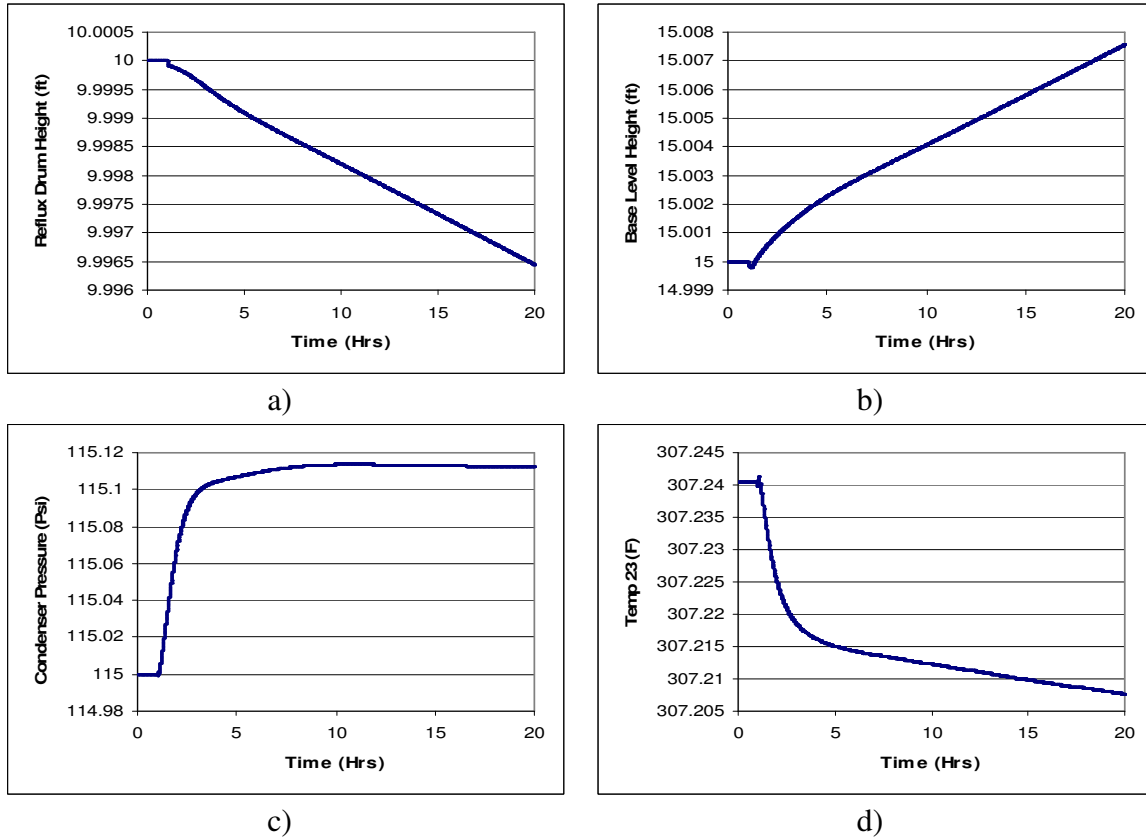
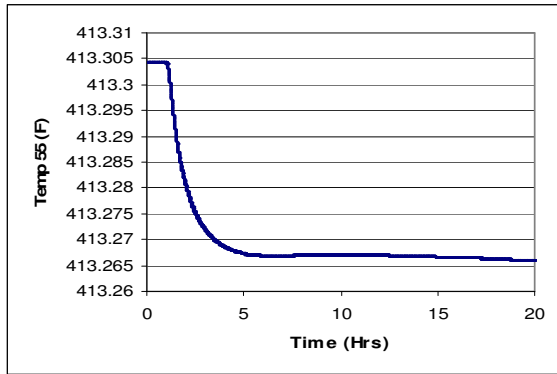


Figure 14: Change in reflux flow rate. Open loop response of: a) reflux drum liquid height b) base level liquid height c) condenser pressure d) temperature on stage 23 e) temperature on stage 55



e)

Figure 14: Continued

An increase in the reflux flow rate shows a decrease in the reflux drum liquid height as expected, while the base level liquid increases. There is a decrease in the temperature in the column as the reflux rate is increased, and more liquid that is coming from the reflux drum cools the column. The response of the pressure and temperatures are first-order process, while the liquid levels have an integrating-process response, which was the case for the most part for the manipulated variables studied.

Getting approximate transfer function

From the data used to create the figures above, the transfer functions were derived using a matlab GUI⁶⁰. The transfer functions between the manipulated and controlled variables are given in tables 3 and 4 below for both control structures.

Table 3: Transfer functions for control structure 1.

	Reflux Drum Height (ft)	Base Level Height (ft)	Condenser Pressure (psi)	Temp 23 (F)	Temp 55 (F)
Bottom Flow Rate	$\frac{-8.68 \times 10^{-7} (-1.232s + 1)e^{-0.554s}}{8.189s + 1}$	$\frac{-8.53 \times 10^{-5}}{s}$	$\frac{-1.04 \times 10^{-4} e^{-1.07s}}{2.437s + 1}$	$\frac{-4.45 \times 10^{-5}}{2.561s + 1}$	$\frac{-4.9 \times 10^{-5}}{2.622s + 1}$
Distillate Flow Rate	$\frac{-4.2 \times 10^{-5}}{s}$	$\frac{2.78 \times 10^{-7} e^{-0.183s}}{s}$	$\frac{-2.57 \times 10^{-4}}{0.5843s + 1}$	$\frac{-2.27 \times 10^{-6} e^{-0.116s}}{s}$	$\frac{-1.58 \times 10^{-5} e^{-0.565s}}{2.654s + 1}$
Vent Flow Rate	$\frac{-1.01 \times 10^{-4}}{s}$	$\frac{1.13 \times 10^{-4} e^{-0.184s}}{s}$	$\frac{-1.05 \times 10^{-1}}{0.582s + 1}$	$\frac{-5.72 \times 10^{-3} e^{-0.245s}}{6.205s + 1}$	$\frac{-6.43 \times 10^{-3} e^{-0.56s}}{2.798s + 1}$
Reboiler Duty	$\frac{1.59 \times 10^{-7} (-1.249s + 1)e^{-0.49s}}{8.249s + 1}$	$\frac{-1.41 \times 10^{-8} e^{-0.978s}}{s}$	$\frac{1.88 \times 10^{-5} e^{-1.07s}}{2.445s + 1}$	$\frac{8.11 \times 10^{-6}}{2.646s + 1}$	$\frac{8.92 \times 10^{-6}}{2.646s + 1}$
Condenser Duty	$\frac{-3.62 \times 10^{-7}}{8.463s + 1}$	$\frac{2.8 \times 10^{-8} e^{-0.325s}}{s}$	$\frac{-3.64 \times 10^{-5}}{1.925s + 1}$	$\frac{-5.99 \times 10^{-6} e^{-0.56s}}{3.505s + 1}$	$\frac{-7.52 \times 10^{-6} e^{-0.545s}}{2.907s + 1}$

Table 4: Transfer functions for control structure 2.

	Reflux Drum Height (ft)	Base Level Height (ft)	Condenser Pressure (psi)	Temp 23 (F)	Temp 55 (F)
Bottom Flow Rate	$\frac{-8.68 \times 10^{-7} (-1.232s + 1)e^{-0.554s}}{8.189s + 1}$	$\frac{-8.53 \times 10^{-5}}{s}$	$\frac{-1.04 \times 10^{-4} e^{-1.07s}}{2.437s + 1}$	$\frac{-4.45 \times 10^{-5}}{2.561s + 1}$	$\frac{-4.9 \times 10^{-5}}{2.622s + 1}$
Distillate Flow Rate	$\frac{-4.2 \times 10^{-5}}{s}$	$\frac{2.78 \times 10^{-7} e^{-0.183s}}{s}$	$\frac{-2.57 \times 10^{-4}}{0.5843s + 1}$	$\frac{-2.27 \times 10^{-6} e^{-0.116s}}{s}$	$\frac{-1.58 \times 10^{-5} e^{-0.565s}}{2.654s + 1}$
Vent Flow Rate	$\frac{-1.01 \times 10^{-4}}{s}$	$\frac{1.13 \times 10^{-4} e^{-0.184s}}{s}$	$\frac{-1.05 \times 10^{-1}}{0.582s + 1}$	$\frac{-5.72 \times 10^{-3} e^{-0.245s}}{6.205s + 1}$	$\frac{-6.43 \times 10^{-3} e^{-0.56s}}{2.798s + 1}$
Reboiler Duty	$\frac{1.59 \times 10^{-7} (-1.249s + 1)e^{-0.49s}}{8.249s + 1}$	$\frac{-1.41 \times 10^{-8} e^{-0.978s}}{s}$	$\frac{1.88 \times 10^{-5} e^{-1.07s}}{2.445s + 1}$	$\frac{8.11 \times 10^{-6}}{2.646s + 1}$	$\frac{8.92 \times 10^{-6}}{2.646s + 1}$
Reflux Flow Rate	$\frac{-5.34 \times 10^{-6}}{s}$	$\frac{1.17 \times 10^{-5}}{s}$	$\frac{3.12 \times 10^{-3} e^{-0.158s}}{0.9306s + 1}$	$\frac{-8.25 \times 10^{-4}}{1.755s + 1}$	$\frac{-1.04 \times 10^{-3}}{0.9472s + 1}$

Relative Gain Array (RGA) analysis

Given the gain of each manipulated variable (input) with corresponding controlled variable (output), we can compute the relative gain array. We place the gain for both control structures in a 5X5 matrix. Since the reactive distillation column is integrating, steady-state gains are not defined for the reflux drum liquid level or for the reboiler liquid volume. However, some information regarding these variables is required for computing the RGA. The approach initially developed by Woolverton⁶¹ and further described by McAvoy⁵⁵ is employed. For variables that exhibit integrating responses, the RGA is calculated based on the derivatives of the variables with respect to time. The gain matrix for both structure are given in the tables 5 and 6:

Table 5: Gain matrix: control structure 1

	Reflux Drum	Base Level	Condenser Pressure	Temp 23	Temp 55
Bottom	-8.67937E-07	-0.001621578	-0.00010501	-4.54E-05	-4.96E-05
Distillate	-0.000806092	5.08228E-06	-0.000259113	-1.42E-05	-1.58E-05
Vent	-0.001892057	0.00209201	-0.105975164	-0.005724	-0.006498
Reboiler	1.58697E-07	-2.52936E-07	1.90273E-05	8.18E-06	9E-06
Condenser	-3.61953E-07	5.21349E-07	-3.69595E-05	-6.08E-06	-7.6E-06

Table 6: Gain matrix: control structure 2

	Reflux Drum Level	Base Level	Condenser Pressure	Temp 23	Temp 55
Bottom	-8.67937E-07	-0.001621578	-0.00010501	-4.54E-05	-4.96E-05
Distillate	-0.000806092	5.08228E-06	-0.000259113	-1.42E-05	-1.58E-05
Vent	-0.001892057	0.00209201	-0.105975164	-0.005724	-0.006498
Reboiler	1.58697E-07	-2.52936E-07	1.90273E-05	8.18E-06	9E-06
Reflux	-9.86584E-05	0.000209569	0.00311582	-0.000906	-0.001064

Performing Relative Gain Array analysis on the gain matrix above, the pairing of variables can be achieved. The relative gain arrays for both control structures are given

in tables 7 and 8 for CS1 and CS2 respectively, with the recommended pairings in bold.

Tables 9 and 10 shows each controlled variable paired with its respective manipulated variable, for both CS1 and CS2 respectively.

Table 7: Relative Gain Array: control structure 1

	Reflux Drum	Base Level	Condenser Pressure	Temp 23	Temp 55
Bottom	-5.3E-08	0.999108	0.00136	0.004437	-0.00491
Distillate	1.005854	-3.1E-07	-0.00637	-0.00394	0.004454
Vent	-0.00597	0.000121	1.21562	1.652735	-1.8625
Reboiler	-4.7E-05	0.000902	-0.11775	7.079145	-5.96225
Condenser	0.000164	-0.00013	-0.09286	-7.73238	8.825206

Table 8: Relative Gain Array: control structure 2

	Reflux Drum	Base Level	Condenser Pressure	Temp 23	Temp 55
Bottom	7.48379E-07	0.999937	0.000821	-0.11351	0.112751
Distillate	1.00486034	3.16E-06	-0.0046	0.045323	-0.04559
Vent	-0.005225767	-0.00034	0.983576	-4.69163	4.71362
Reboiler	-0.000124644	0.000971	-0.06571	18.40463	-17.3398
Reflux	0.000489323	-0.00058	0.085908	-12.6448	13.55899

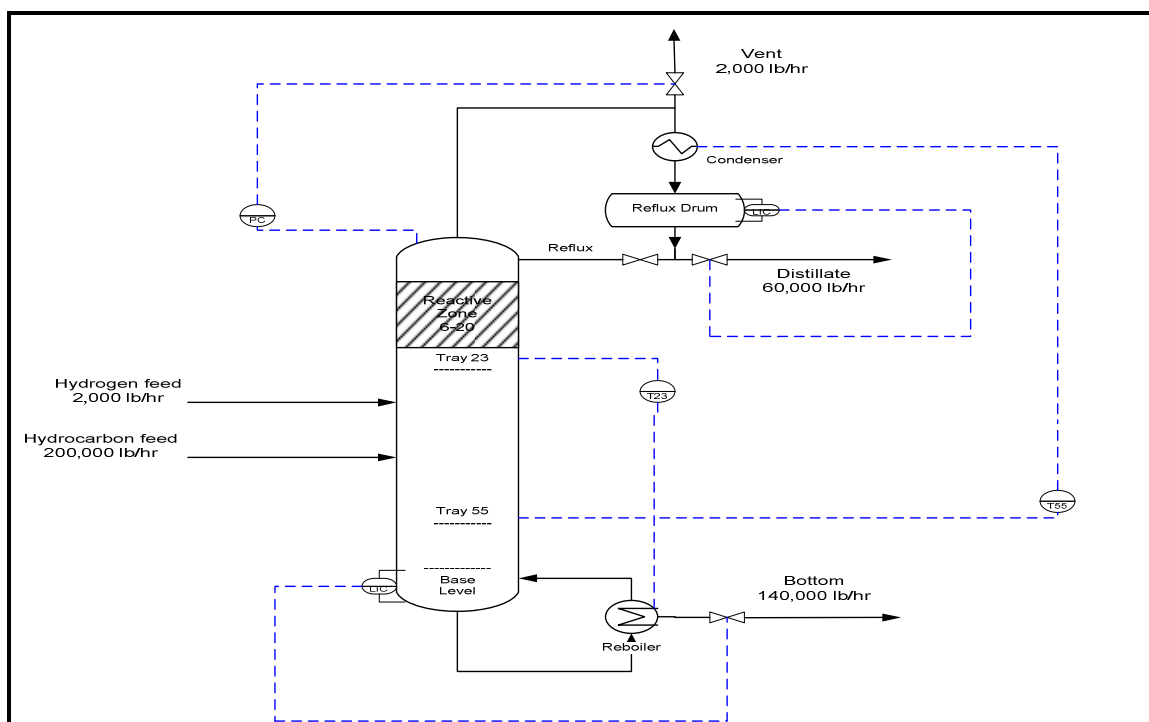
Table 9: Controlled variable paired with corresponding manipulated variable for CS1

Controlled Variable	Manipulated Variable
Base level height	Bottom flow rate
Reflux drum level height	Distillate flow rate
Condenser pressure	Vent flow
Temperature (Stage 23)	Reboiler duty
Temperature (Stage 55)	Condenser duty

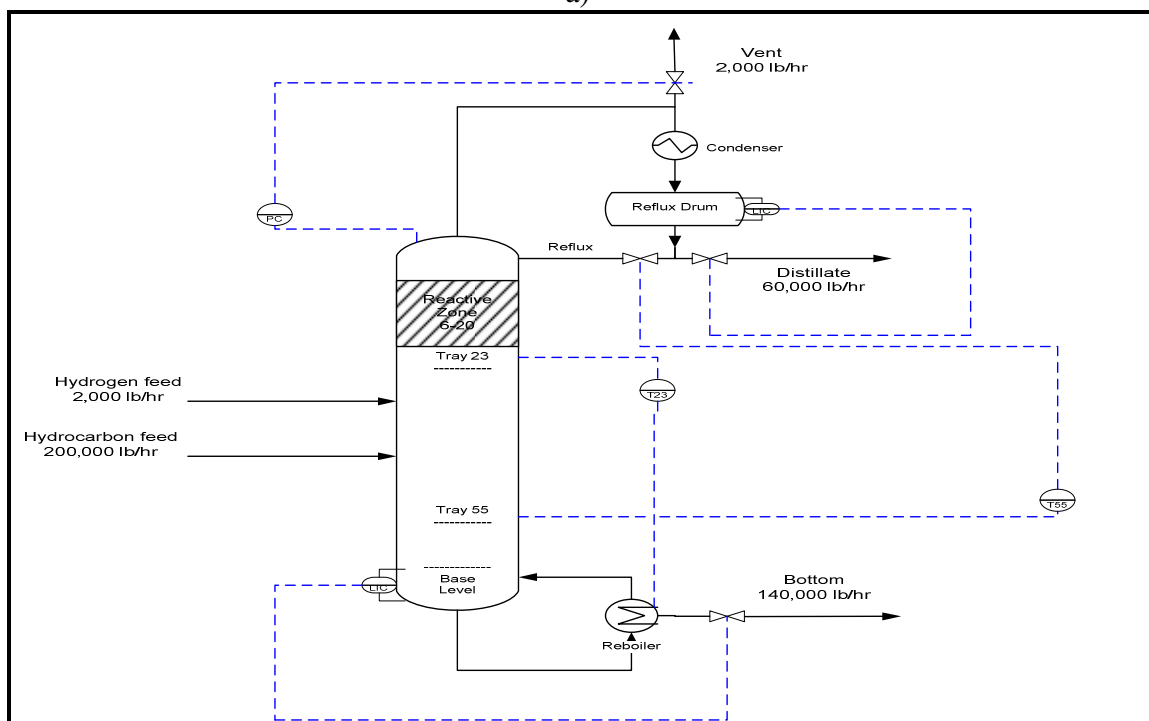
Table 10: Controlled variable paired with corresponding manipulated variable for CS2

Controlled Variable	Manipulated Variable
Base level height	Bottom flow rate
Reflux drum level height	Distillate flow rate
Condenser pressure	Vent flow
Temperature (Stage 23)	Reboiler duty
Temperature (Stage 55)	Reflux flow rate

According to Bristol's recommendation for controller pairing, controlled variables and manipulated variables are paired so that the corresponding relative gains are positive and as close to one as possible. From the above table, the two control structures have similar controller pairing except for the difference of the manipulated variables used for controlling the temperature on stage 55. From the controller pairing and the transfer function the controllers can now be tuned. Figure 15 shows the process flow diagram of the two control structures.



a)



b)

Figure 15: Process flow diagram. a) Control structure 1 b) Control structure 2

CHAPTER V

CONTROL STRUCTURE OF REACTIVE DISTILLATION WITH PI CONTROLLERS

Controller tuning

Before the controllers are implemented, they have to be tuned to provide good controller performance for the dynamic process. From the pairing recommended by the RGA analysis, we have 5 controllers to tune and from the transfer function for paired variables we can get an initial value of the controller parameters by using IMC tuning method. Since the process is simulated in Aspen Dynamics, the units of the gains have to be converted from the respective units to (%/%). The gain, time constant and time delay are then used to calculate the controller gain and reset time. The choice of the desired closed-loop time constant has to be taken into consideration, because it is used to calculate the controller parameters. Choosing a small desired closed-loop time constant will lead to a more aggressive controller that would return the process back to its steady state within a considerably shorter amount of time than choosing a larger time constant. Also, we do not want to choose a very small desired closed-loop time constant that will lead to the system being oscillatory and unstable when a disturbance is charged into the system. There are several rules of thumbs in choosing a desired closed-loop time constant⁵⁷. Tables 11 and 12, show the parameter for each controller for both control structure. As a rule of thumb, the desired closed loop constant should be smaller than the open-loop time constant and greater than the delay time: $\tau > \tau_c > \theta$.⁶²

Table 11: Control settings for control structure 1 using IMC tuning rules

	Reflux Level Controller	Base Level Controller	Pressure Controller	Stage 23 Temperature Controller	Stage 55 Temperature Controller
Controller gain (%/%)	70.73	23.77	50.00	13.24	90.00
Reset time (hr)	0.22	0.22	0.001	0.10	0.25

Table 12: Control settings for control structure 2 using IMC tuning rules

	Reflux Level Controller	Base Level Controller	Pressure Controller	Stage 23 Temperature Controller	Stage 55 Temperature Controller
Controller gain (%/%)	74.28	24.16	50.00	8.76	10.00
Reset time (hr)	0.22	0.22	0.001	0.11	0.06

The control structure is implemented and the performance is investigated.

Disturbance rejection is investigated for each controller in both control structures.

Disturbances charged to the column are feed flow rate, feed temperature and feed composition.

Control performance

Table 13: Disturbances to system

Perturbation	Description
D ₁	Reformat feed flow rate +5%
D ₂	Feed Temperature +10%
D ₃	Feed benzene composition from 0.08 to 0.09

For the perturbations in Table 13, closed-loop responses are simulated in Aspen

Dynamics and the control performance of both structures are assessed and compared.

Figure 16 shows the closed loop response of both control structures to disturbance in the feed flow rate.

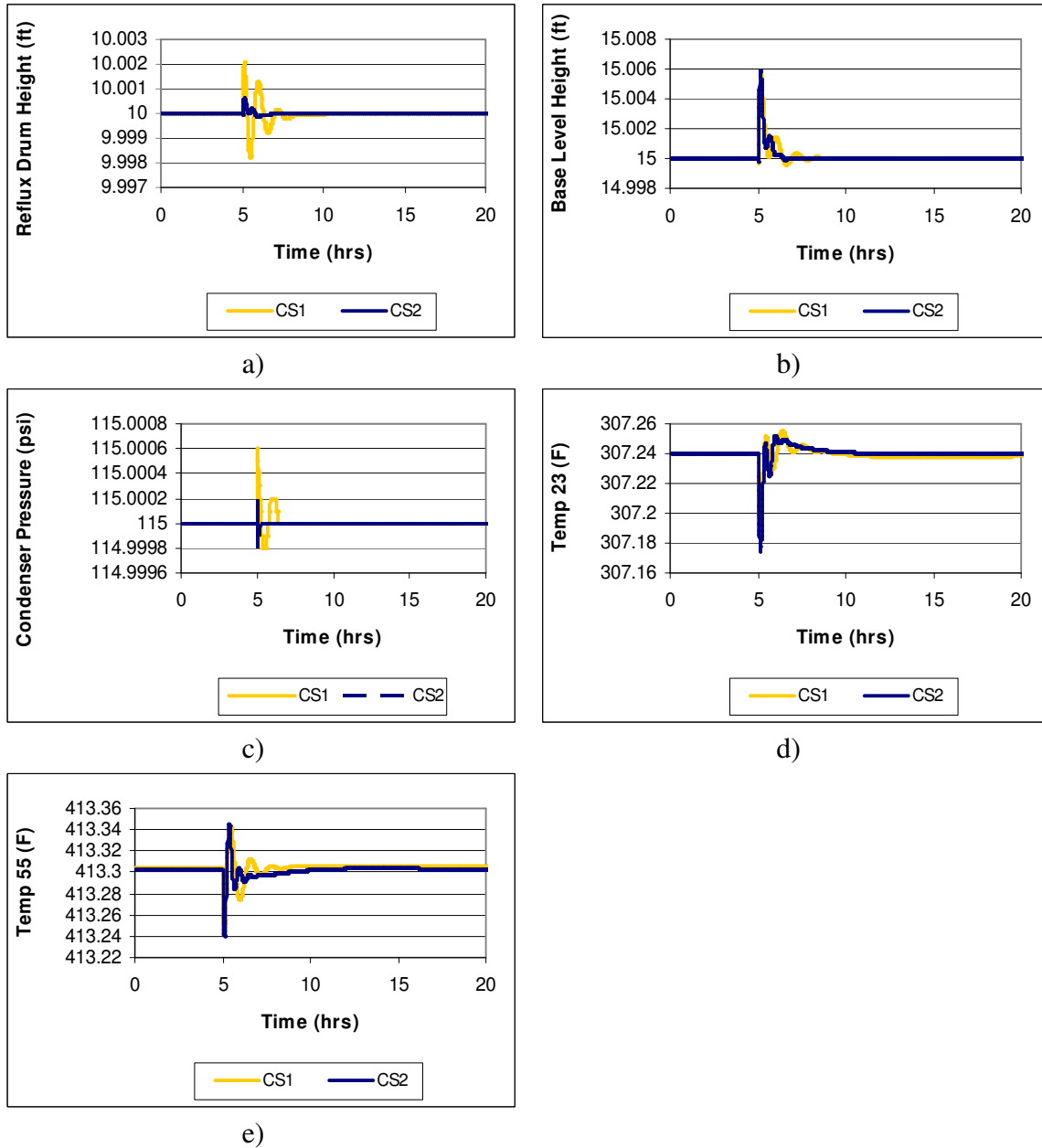


Figure 16: Feed flow rate disturbance (D_1). Closed loop response to a) reflux drum liquid height b) base level liquid height c) condenser pressure d) temperature on stage 23 e) temperature on stage 55

Figure 17 below shows the closed loop response of both control structures to disturbances in feed temperature.

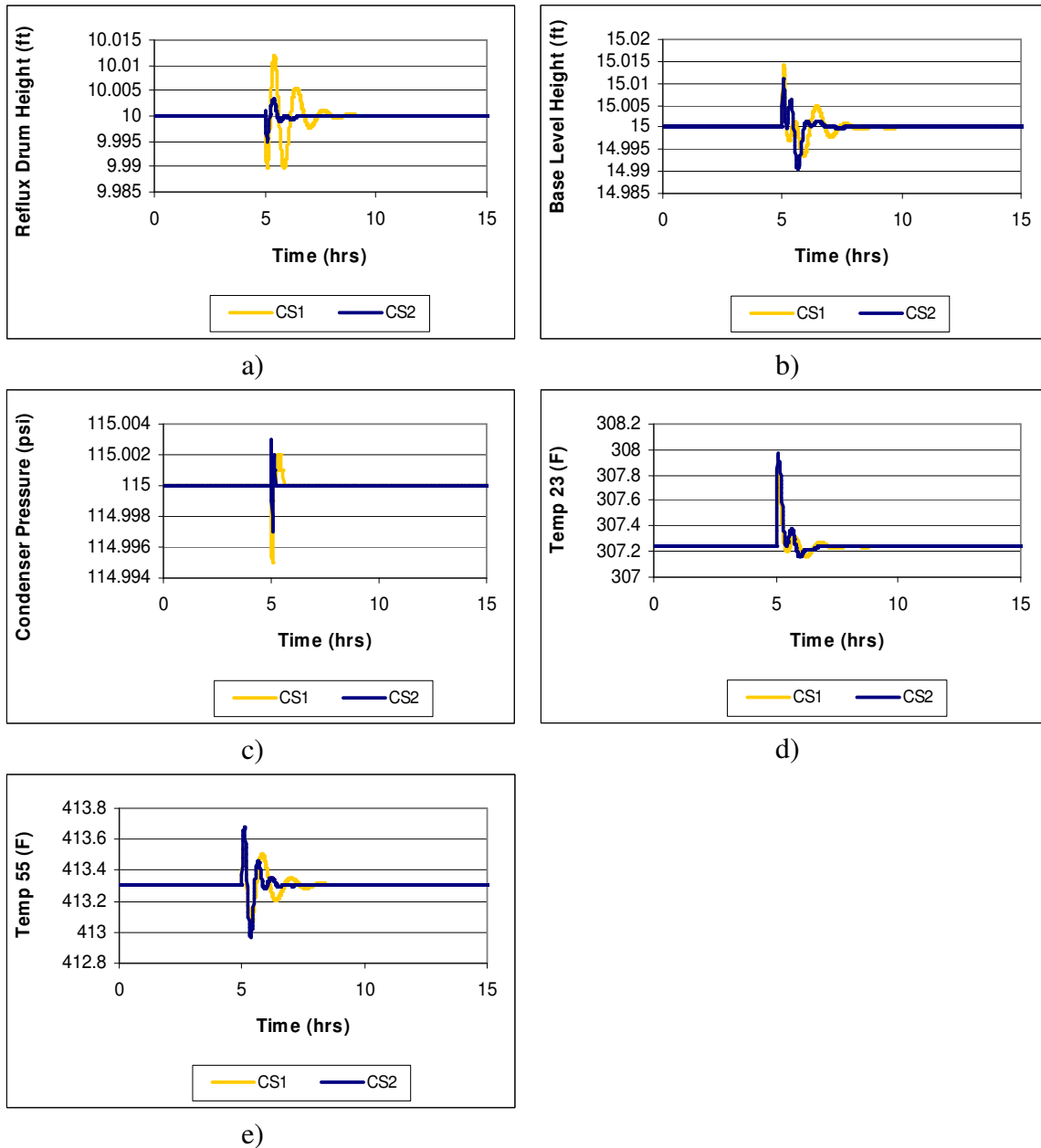


Figure 17: Feed temperature disturbance (D_2). Closed loop response to a) reflux drum liquid height b) base level liquid height c) condenser pressure d) temperature on stage 23 e) temperature on stage 55

Figure 18 shows the closed loop response of both control structures to disturbances in feed benzene composition.

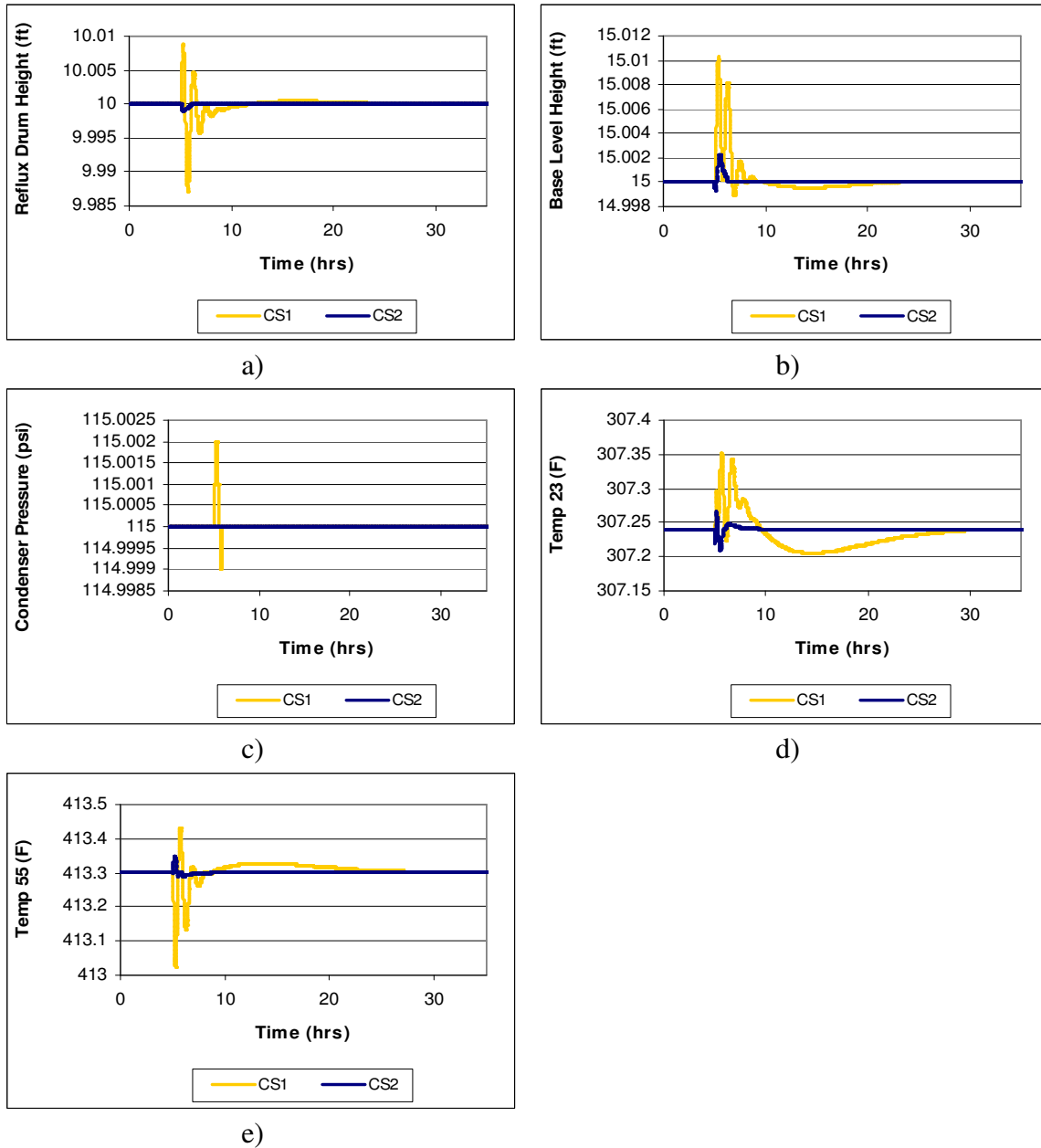


Figure 18: Feed benzene composition disturbance (D_1). Closed loop response to a) reflux drum liquid height b) base level liquid height c) condenser pressure d) temperature on stage 23 e) temperature on stage 55

Discussion

The controller settings above give a satisfactory controller performance. From the figures above, the controllers are returning each controlled variable back to steady state in less than 5 hrs. Control structure 2 generally performs better than control structure 1, it is less oscillatory and it gets back to steady state faster. Perturbation (D_3) is where the advantage of CS2 over CS1 is emphasized. It takes CS1 about 25 hours to return to steady state after a feed composition disturbance, whereas CS2 returns the column to steady state in approximately 3 hours.

CHAPTER VI

CONCLUSIONS AND RECOMMENDATIONS

Conclusions

The dynamic model of a reactive distillation column for benzene hydrogenation was created and investigated. The column has 70 trays and is fed with a stream that contains 12 components. Hydrogen was fed above the hydrocarbon feed stage to hydrogenate the benzene in the process. The feed to the system comes from a reformer and may change on a regular basis which presents a need for investigating the dynamic behavior of the process. EPA regulations require that the benzene content in the distillate streams leaving the column is below 1% weight concentration even under the influence of large disturbances in the feed. Building a dynamic model started by creating a steady state model for the process. The results from the steady state simulation were compared with data provided from an industrial collaborator. The steady state results were then extended to be used to create the dynamic model, which is used to design the control structure for the process.

Open loop tests were performed on the dynamic model for different disturbances and the need for the work was revealed as the process moves away from its steady state, hence the need for a control system. A set of manipulated and controlled variables were chosen and open loop simulations were carried out to determine the transfer functions between the variables.

A 5x5 control structure was implemented on the process and investigated. Two control structures were compared and analyzed. RGA analysis was performed on the dynamic model to find optimal pairing for the control structures. Naturally, the RGA analysis suggested pairing the condenser pressure with the vent flow rate, the base level liquid height with the bottom flow rate and the reflux drum liquid level with the distillate flow rate. The three pairing are in agreement with studies dedicated to distillation or reactive distillation.^{24, 63} The other two variables are used to control the temperature on two different stages (stage 23 and 55).

PI controllers were designed and tuned based upon the transfer function between the manipulated and the controlled variable and the IMC tuning rules were used to obtain the controller settings. The controllers provided satisfactory control performance, where all the control variables return to steady state within 5 hours of when disturbance was charged.

From our results, control structure 2 performed better than control structure 1. The main advantage of CS2 over CS1 is noticed in the simulation of feed composition disturbance rejection, where CS2 returns all variables back to steady state within 3 hrs while it take CS1 more than 20 hrs to return the temperature variables back to steady state. This means, it would better to control the temperature on stage 55 with the reflux flow rate rather than the condenser duty.

There is a possibility the control performance can be further improved by investigating feed forward and cascade control and also trying to implement model predictive control (MPC) for the column.

This is one of the first works on the control of a reactive distillation for benzene hydrogenation, in our approach we used temperature controllers as opposed to conventional composition controllers used in simulation but hardly used in real-life situations.

Future work

The 5x5 control of a benzene hydrogenation reactive distillation column was investigated however supplemental research and simulations can be done to extend the work done here. The following issues are worth of investigation:

- Choosing different sets of manipulated and controlled variables to investigate if it gives any advantage to the existing control structure developed.
- Feed forward control studies can be implemented on the process, to investigate if it improves the process controllability.
- Cascade control structures can also be investigated on the process.
- Also implementing model predictive control (MPC) on the column may have its advantages, so it should be taken into consideration for investigation.

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